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Evaluation & Comparison of the Cobalt Thiocyanate & Mod. Dragendorf Reagent Test Methods for Nonionic Detergents

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EVALUATION AND COMPARISON OF THE COBALT THIOCYANATE AND MODIFIED DRAGENDORF REAGENT TEST METHODS FOR NONIONIC DETERGENTS

Henry H. Tabak, Robert N. Bloomhuff and Robert L. Bunch

Environmental Protection Agency
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Studies were undertaken to evaluate a modified Dragendorf reagent test method for the determination of nonionic surfactants in influents and effluents of biological wastewater treatment plants and to compare its sensitivity with that of the cobalt thiocyanate method for nonionic detergents. The WICKBOLD method is based on the precipitation of the surfactant with a modified Dragendorf reagent (K_2BiI_4 + $BaCl_2$ - + acetic acid). The filtered precipitate is washed with acetic acid and dissolved in ammonium tartrate solution. The bismuth from the bismuth-surfactant complex in solution is than titrated potentiometrically at pH 4 - 5 with 0.0005 N pyrrolidine dithiocarbamate solution using a platinum and calomel electrode. The cobalt thiocyanate method involves the treatment of the detergent with ammonium cobalt thiocyanate solution, chloroform extraction of the surfactant-cobalt thiocyanate complex and the spectrophotometric analysis of the complex in chloroform at 318 nm. The three nonionic surfactants used in the studies were: (1) fatty alcohol ethoxylate with 30.7 ethylene oxide units, (2) fatty alcohol ethoxylate with 10.6 ethylene oxide units, and (3) nonyl phenol ethoxylate,

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*Detergents
*Surfactants,

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Industrial waste treatment,
Precipitation,
Solutions,
Cobalt inorganic compounds,
Thiocyanates,
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EVALUATION AND COMPARISON OF THE COBALT THIOCYANATE AND MODIFIED DRAGENDORF REAGENT TEST METHODS FOR NONIONIC DETERGENTS

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Studies were undertaken to evaluate a modified Dragendorf reagent test method[1-3] for the determination of nonionic surfactants in influents and effluents of biological wastewater treatment plants and to compare its sensitivity with that of the cobalt thiocyanate method[4] for nonionic detergents.

The WICKBOLD (3) method is based on the precipitation of the surfactant with a modified Dragendorf reagent (K_2BiI_4 + $BaCl_2$ + acetic acid). The filtered precipitate is washed with acetic acid and dissolved in ammonium tartrate solution. The bismuth from the bismuth-surfactant complex in solution is then titrated potentiometrically at pH 4 - 5 with 0.0005 N pyrrolidine dithiocarbamate solution using a platinum and calomel electrode.

The cobalt thiocyanate method involves the treatment of the detergent with ammonium cobalt thiocyanate solution, chloroform extraction of the surfactant-cobalt thiocyanate complex and the spectrophotometric analysis of the complex in chloroform at 318 nm.

The three nonionic surfactants used in the studies were: (1) fatty alcohol ethoxylate with 30.7 ethylene oxide units, (2) fatty alcohol ethoxylate with 10.6 ethylene oxide units, and (3) nonyl phenol ethoxylate with 9.0 ethylene oxide units. In the cobalt thiocyanate method studies the stock solutions of the surfactants were made up in chloroform and in distilled water.

The chloroform solutions of the detergents were used in tests with unextracted samples, whereas the aqueous stock solutions were employed in tests for surfactants extracted from water system. In the modified Dragendorf reagent method studies, the stock solutions of the surfactants were made up in methyl alcohol, and were employed in tests with unextracted samples and with surfactants extracted from water and wastewater systems.

In the studies with the Cobalt Thiocyanate Method, chloroform was used as the solvent in extraction of surfactants from water system. Ethyl acetate was employed as the solvent for extracting the surfactant from water and wastewater in studies with the modified Dragendorf Reagent Method. Potentiometric titrations were accomplished with the use of the Corning Research pH Meter, Varian Recorder with 10 mv span and Technicon Autoanalyzer proportioning pump.

The results with the cobalt thiocyanate method indicate good sensitivity in the range of 100 to 1,000 μg of each of the three nonionic surfactants. The degree of sensitivity increases with the increase in the number of the ethylene oxide units on the surfactant molecule. Standard curves were produced for each of the three surfactants. The absorbance data for the unextracted surfactants are presented in Tables 1 through 3. The calibration curves for the same samples are expressed in Figures 1 through 3. The slopes decrease in values with the decrease in number of the ethylene oxide units on the molecules, and thus the fatty alcohol with 30.7 ethylene oxide units is expressed by a standard curve with relatively higher slope values than those of the standard curves for the fatty alcohol with 10.6 moles and nonyl phenol ethoxylate with 9.0 moles.

The sensitivity decreases slightly with the extracted surfactants from aqueous media because the percent recovery for each of the compounds was in the range of 90 to 95 percent. The absorbence data for extracted detergents are shown in Tables 4 through 6, and the calibration curves of the extracts are expressed in Figures 4 through 6. There is a linear relationship between the concentration of the extracts of each of the surfactants and the absorbence, for the range of concentrations between 100 and 1,000 μg.

Experiments with the modified Dragendorf reagent test involving potentiometric titrations of bismuth from bismuth-surfactant complex with pyrrolidine dithiocarbamate provided analytical data which reveal a high sensitivity of the method. The new method was shown to be very applicable in quantitating the polyoxyethylene nonionic surfactants both in distilled water system, untreated wastes, and in plant effluent.

The potentiometric titration data for the unextracted polyoxyethylene nonionic surfactants are presented in Tables 7 through 9. The reproducibility of the values for each concentration of the surfactant is adequately demonstrated for three separate experiments. A very significant linear relationship is evidenced between potentiometric break distance values in chart units and volumes of the titrant, pyrrolidine dithiocarbamate used for each surfactant concentration in the range between 50 to 1,000 μg of the compound.

The potentiometric break distances represent distances in chart units from the point at which titration was initiated to the point of inflection of the curve (the point at which there is exhibited the greatest rate of loss of potential in mv per each chart distance unit, or the greatest change in y axis per unit of x axis).

The calibration curves for the three unextracted surfactants expressing the relationship between the concentration of the detergent and potentiometric break distance are presented in Figures 7 through 9, while those relating the surfactant concentration to the volume of pyrrolidine dithiocarbamate are shown in Figures 10 through 12. The standard curves demonstrate a linear relationship between the concentrations of each of the three compounds and the potentiometric break distances or volumes of T-Carbat used, within the concentration range from 50 to 1,000 μg. The slopes decrease in value as the number of ethylene oxide units on the surfactant molecule decreases, and this trend is represented in Figures 7 through 9 by the lowest standard curve slope (5.0) for fatty alcohol ethoxycohol ethoxylate 30.7 moles, a medium standard curve slope (6.4) for fatty alcohol ethoxylate 10.6 moles and the highest curve slope (7.2) for nonyl phenol ethoxylate 9.0 moles.

In the extraction procedure, the blow-off method was substituted by solvent extraction in 250 ml capacity separatory funnels on reciprocal shakers. The appropriate concentrations of each of the nonionic detergents were added to 100 ml volumes of either distilled water or wastewater in separatory funnels and the contents were extracted three times with 50 ml volumes of ethyl acetate. The solvent fractions were pooled into evaporation flasks and the solvent was evaporated with the use of a flash evaporator to dryness. The dry residue was then dissolved in water and methanol and the sample was subjected to the test method. The modification of the extraction procedure proved to be efficient enough to give a high percent recovery of the surfactant and the high recovery values were observed at all concentrations of the surfactant used.

The potentiometric titration data for the three nonionic surfactants extracted from water system are shown in Tables 10 through 12. The percent recovery of each of the surfactants was determined to be in the range of 90 to 95 percent of initial concentration. The high recovery values were observed at all concentrations of the surfactant used.

The calibration curves for the polyoxyethylene nonionic surfactants extracted from water system and expressing the relationship between the concentration of the detergent and potentiometric break distance are presented in Figures 13 through 15, and those relating the surfactant concentration to the volume of the titrant are shown in Figures 16 through 18. The standard curve slopes for fatty alcohol ethoxylate 30.7 moles, fatty alcohol ethoxylate 10.6 moles and nonyl phenol ethoxylate 9.0 moles were 5.5, 6.7 and 7.5 respectively. The titration end points were relatively sharp, being approximately 20 to 40 mv per chart unit.

The potentiometric titration data from the studies with wastewater reveal good sensitivity in the range of 50 to 1,000 μg. Very little interference from other organic components in wastewater was detected during ethyl acetate extraction and titration. The titration results from the studies with the three surfactant extracted from wastewater are presented in Tables 13 through 15. The average recovery values of the surfactants were in the range of 80 to 90 percent at all concentration levels. The reproducibility of the potentiometric break distance and volume of titrant values is again adequately demonstrated. The relationship between the potentiometric break distances and titrant volumes was observed to be linear in the 50 to 1,000 μg concentration range.

The calibration curves for the three nonionic surfactants extracted from wastewater system and expressing the relationship between the concentration of the detergent and potentiometric break distance are shown in Figures 19 through 21, while those relating the surfactant concentration to the volume of T-Carbat are presented in Figures 22 through 24. The standard curve slopes for fatty alcohol ethoxylate 30.7 moles, fatty alcohol ethoxylate 10.6 moles and nonyl phenol ethoxylate 9.0 moles were shown to be 5.8, 6.9 and 8.0 respectively. The titration end points were again shown to be relatively sharp and in the range of 20 to 40 mv per chart unit.

Throughout the potentiometric titration studies the initial potential of the solution, prior to titration, varied somewhat depending on the concentration of the surfactant. When surfactant levels, less than 300 μg were examined, the initial potentials were observed to be in the range of +240 to +260 mv. Subsequently with time an increase of mv potential was observed until it reached equilibrium at about +285 mv. The titrations were initiated only after the potential of the solution came to an equilibrium. When surfactant levels, more than 300 μg, were examined, the initial potentials were observed in the range of +300 to +320 mv depending on the concentration of the surfactant. Subsequently with time a decrease in mv potential was observed until it reached an equilibrium at about +285 to +290 mv at which time the titration was started. The potentiometric breaks during titration generally occurred in the +260 to +280 region. In general, for all surfactant levels, the titration was usually started in the +285 to +290 mv region (region of equilibrium potential). Millivolt changes in the range of 20 to 40 mv per chart unit were observed in the studies.

The following modifications of the method were introduced to achieve high potentiometric sensitivity and reproducibility of results: (1) Filtration of the precipitate with the use of Gooch crucibles containing inserted glass fiber filters of grade 934 AH. (2) Elimination of excessive use of glacial acetic acid to wash the precipitate to prevent loss of bismuth from test solution. (3) Reduction of excessive volume dilution of the sample with water before titration to increase the sharpness of the potentiometric break. (4) Substitution of concentrated NH_4OH for 1 percent ammonia solution for pH adjustments to prevent needless dilution of sample before titration. (5) Preparation of fresh pyrrolidine dithiocarbamate solution every week and daily standardization with copper sulfate, since it was shown in our studies that within a ten day period there is a 5 to 6 percent drop in titrant strength.

The Wickbold method would be useful only to determine primary biodegradation. That is biodegradation to the minimum extent necessary to change the molecule so that the test procedures do not detect the intact compound. It does not, however, give any information on how far the biodegradation has proceeded. Large amounts of intermediate biodegradation products could go undetected if they are more resistant than the parent compound. The likely appearance of some intermediate products that would become water pollutants must be a factor in examining a compound for acceptability to the environment.

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Table 1 Spectrophotometric Analysis of Unextracted Fatty Alcohol Ethoxylate 30.7 moles EO.

Surfactant Concentration ug	Absorbance			Average Absorbance
	Experiment Number			
	1	2	3	
100	.063	.065	.062	.063
200	.107	.103	.104	.105
300	.164	.161	.163	.163
400	.220	.219	.221	.220
500	.254	.257	.255	.255
600	.307	.312	.310	.310
700	.355	.357	.369	.359
800	.410	.413	.411	.411
900	.470	.472	.470	.471
1,000	.525	.521	.523	.523

Table 2 Spectrophotometric Data on Unextracted Fatty Alcohol Ethoxylate
10.6 moles EO.

Surfactant Concentration ug	Absorbance			Average Absorbance
	Experiment Number			
	1	2	3	
100	.036	.032	.034	.034
200	.080	.075	.077	.077
300	.118	.115	.117	.117
400	.164	.169	.168	.167
500	.200	.198	.201	.200
600	.234	.242	.236	.237
700	.282	.280	.280	.281
800	.332	.338	.335	.335
900	.375	.370	.371	.372
1,000	.421	.427	.423	.424

Table 3 Spectrophotometric Data on Unextracted Nonyl Phenol Ethoxylate
9.0 moles EO.

Surfactant Concentration ug	Absorbance			Average Absorbance
	Experiment Number			
	1	2	3	
100	.030	.034	.033	.032
200	.065	.061	.062	.063
300	.093	.092	.095	.093
400	.130	.124	.125	.126
500	.158	.151	.153	.154
600	.183	.188	.185	.185
700	.222	.225	.223	.223
800	.245	.249	.247	.247
900	.287	.283	.284	.285
1,000	.315	.318	.316	.316

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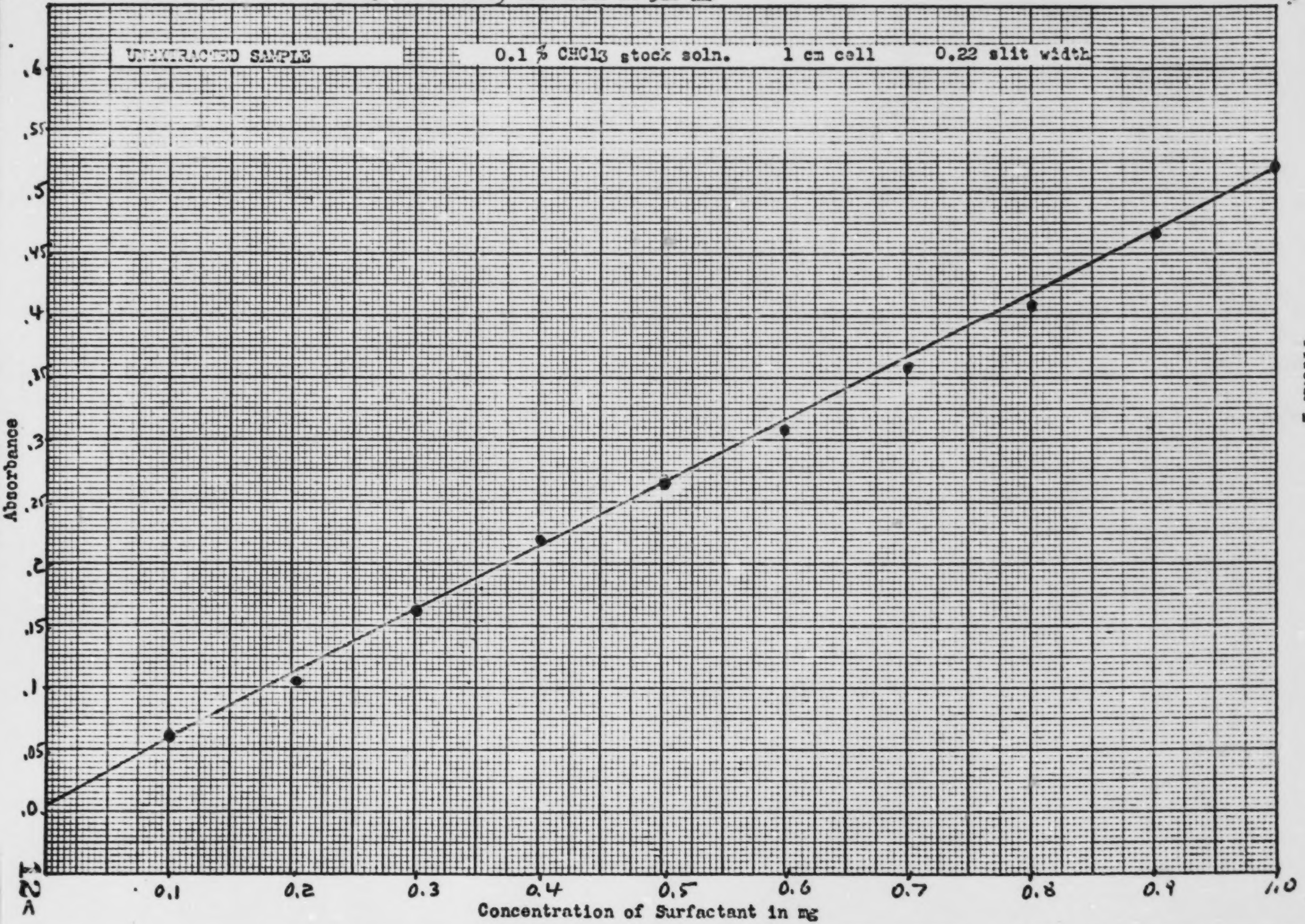
FIGURE 1

STANDARD CURVE Fatty Alcohol Ethoxylate 30.7 moles EO. Cobalt Thiocyanate Method for Non-Ionic Detergents

Spectrophotometric Analysis of $CHCl_3$ solutions at 318 nm



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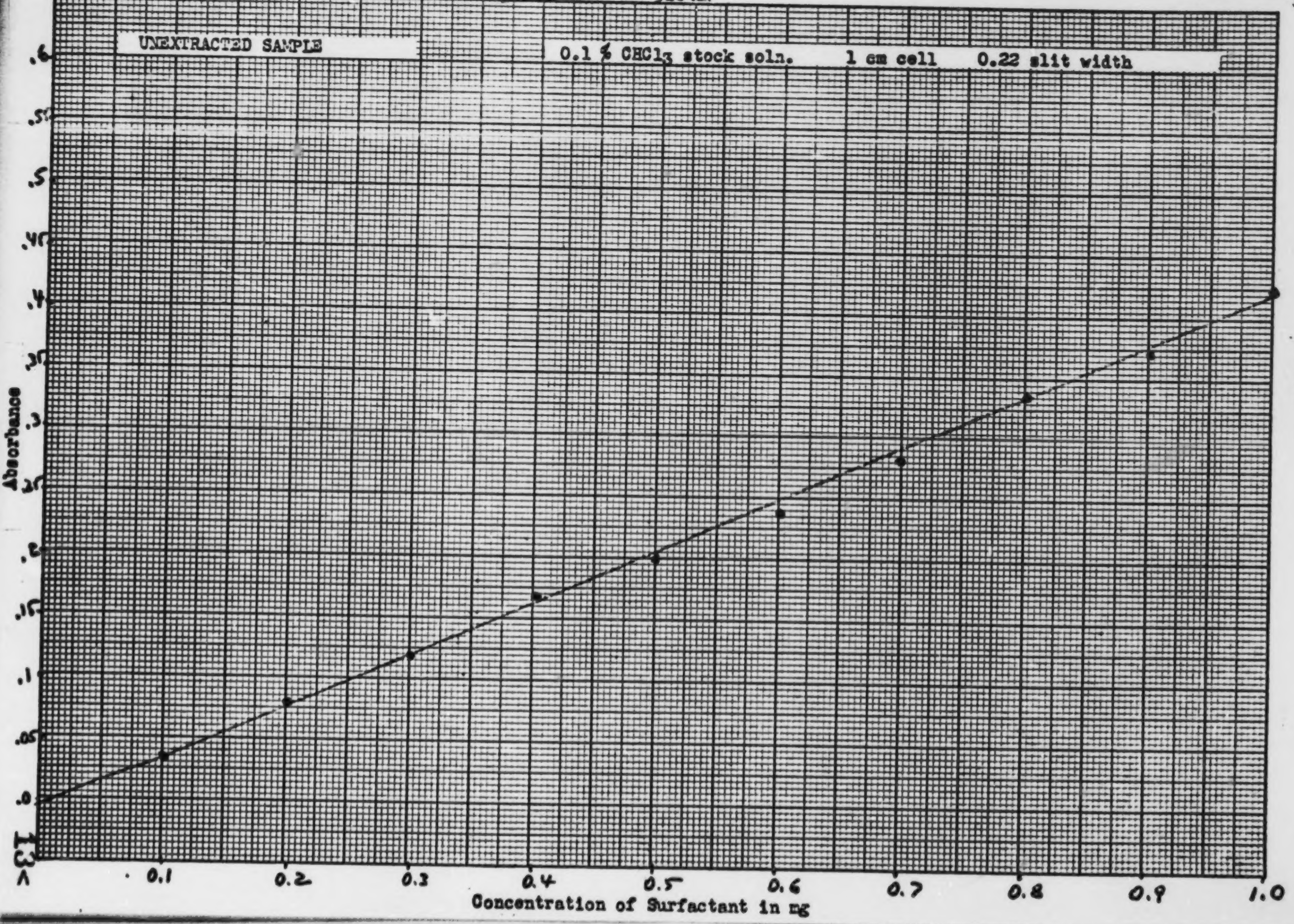

FIGURE 2

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STANDARD CURVE Nonyl Phenol Ethoxylate 9.0 moles EO. Cobalt Thiocyanate Method for Non-Ionic Detergents

Spectrophotometric Analysis of $CHCl_3$ solutions at 318 nm

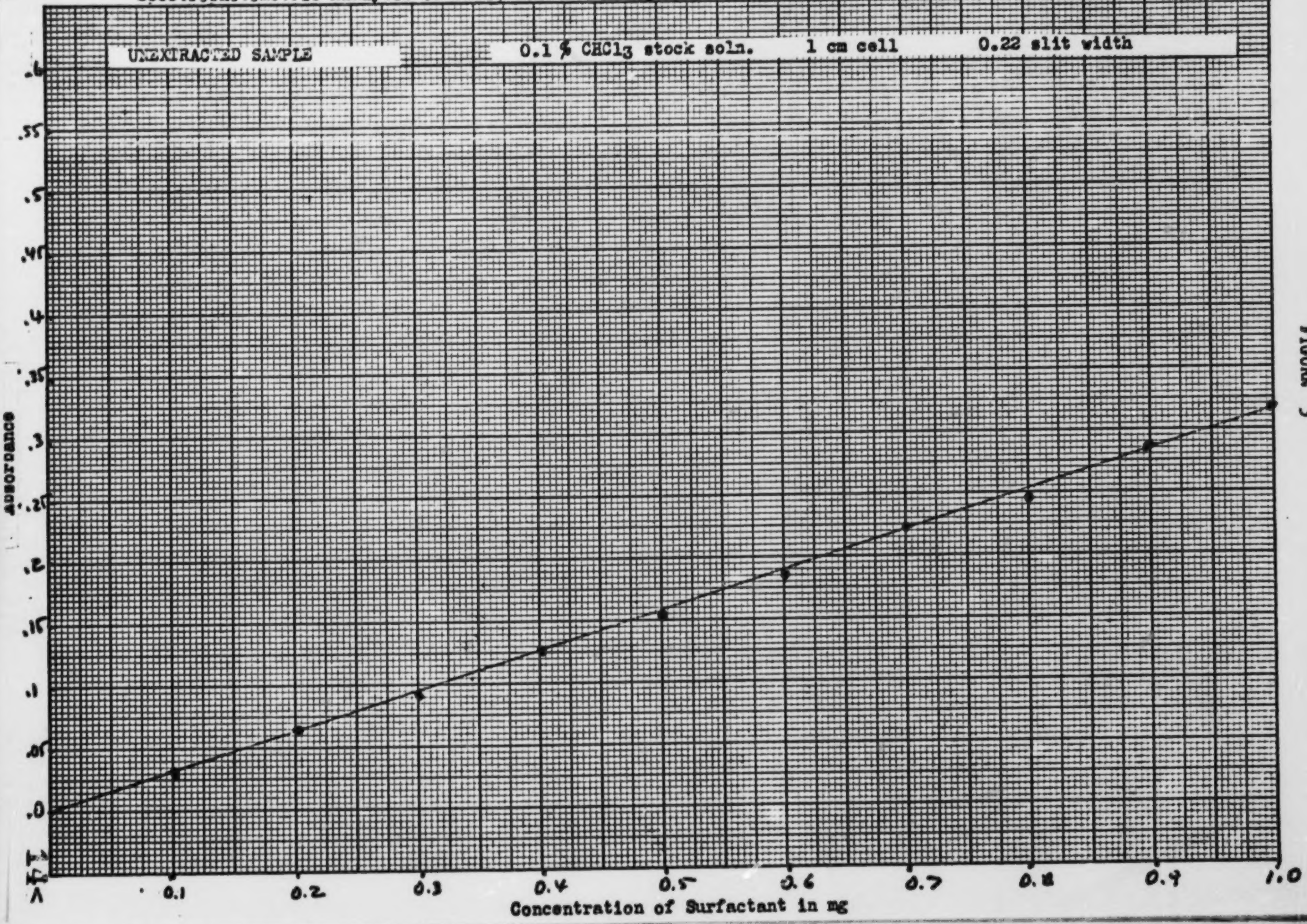

FIGURE 3

Table 4 Spectrophotometric Data on Fatty Alcohol Ethoxylate 30.7 moles EO Extracted from Water System

Surfactant Concentration ug	Absorbance			Average Absorbance
	Experiment Number			
	1	2	3	
100	.057	.055	.053	.055
200	.098	.100	.101	.100
300	.147	.143	.144	.145
400	.192	.187	.188	.189
500	.227	.243	.236	.235
600	.285	.285	.289	.286
700	.325	.329	.325	.326
800	.380	.375	.377	.377
900	.423	.426	.423	.424
1,000	.467	.475	.471	.471

Table 5 Spectrophotometric Data on Fatty Alcohol Ethoxylate 10,6 moles EO. Extracted from Water System

Surfactant Concentration ug	Absorbance			Average Absorbance
	Experiment Number			
	1	2	3	
100	.040	.040	.042	.041
200	.068	.075	.070	.071
300	.100	.110	.103	.104
400	.143	.147	.142	.144
500	.170	.173	.171	.171
600	.205	.215	.208	.209
700	.345	.237	.239	.240
800	.278	.285	.281	.281
900	.313	.314	.318	.315
1,000	.347	.355	.351	.351

Table 6 Spectrophotometric Data on Nonyl Phenol Ethoxylate 9.0 moles EO. Extracted from Water System

Surfactant Concentration ug	Absorbance Experiment Number 1	 2	 3	Average Absorbance
100	.028	.023	.026	.026
200	.050	.057	.051	.053
300	.077	.078	.074	.076
400	.105	.100	.102	.102
500	.120	.125	.126	.124
600	.158	.150	.153	.154
700	.175	.180	.179	.178
800	.208	.200	.202	.203
900	.225	.231	.229	.228
1,000	.255	.255	.258	.256

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Spectrophotometric Analysis of $CHCl_3$ solutions at 318 nm

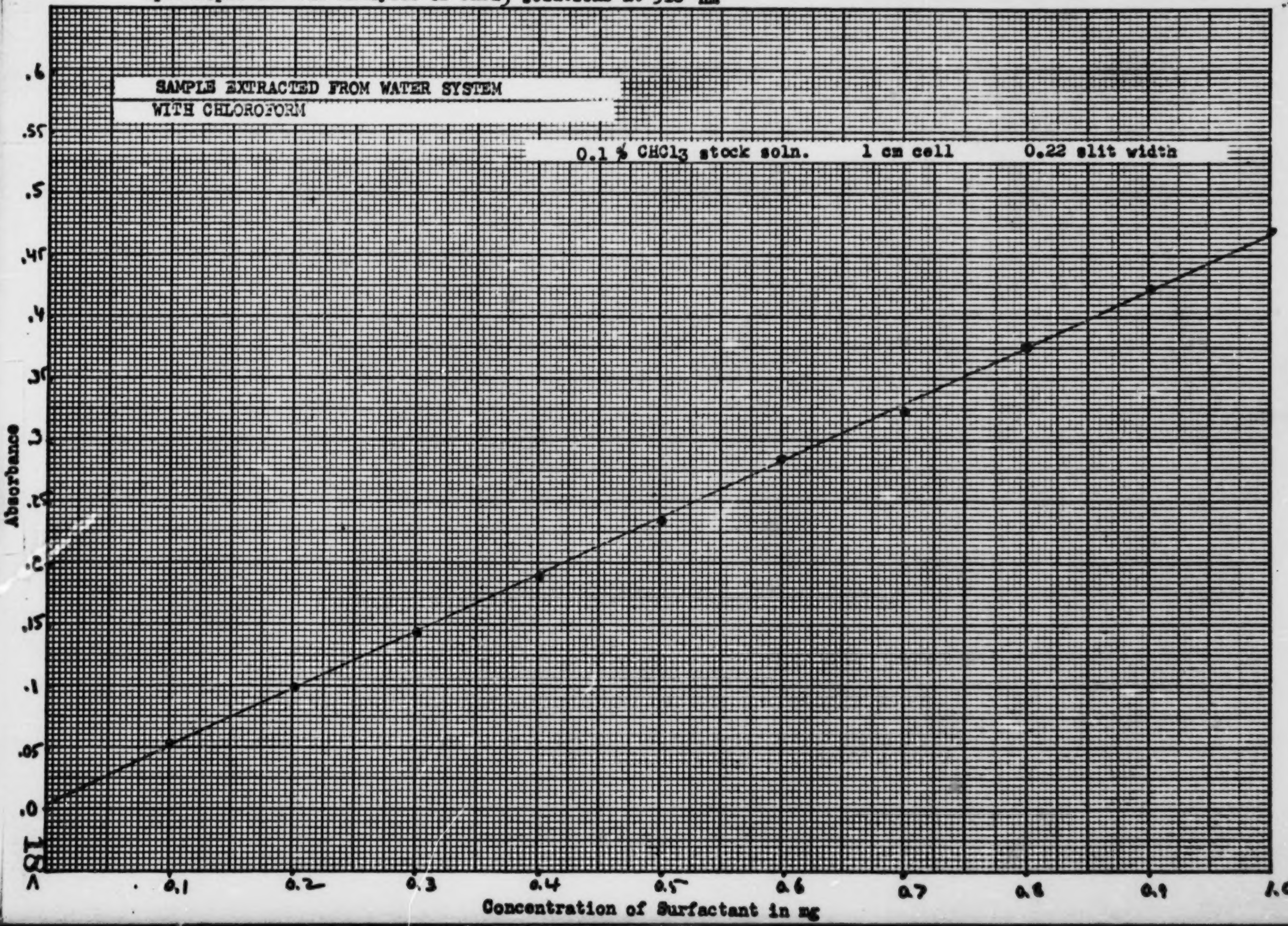

FIGURE 4

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Spectrophotometric Analysis of $CHCl_3$ solutions at 318 nm

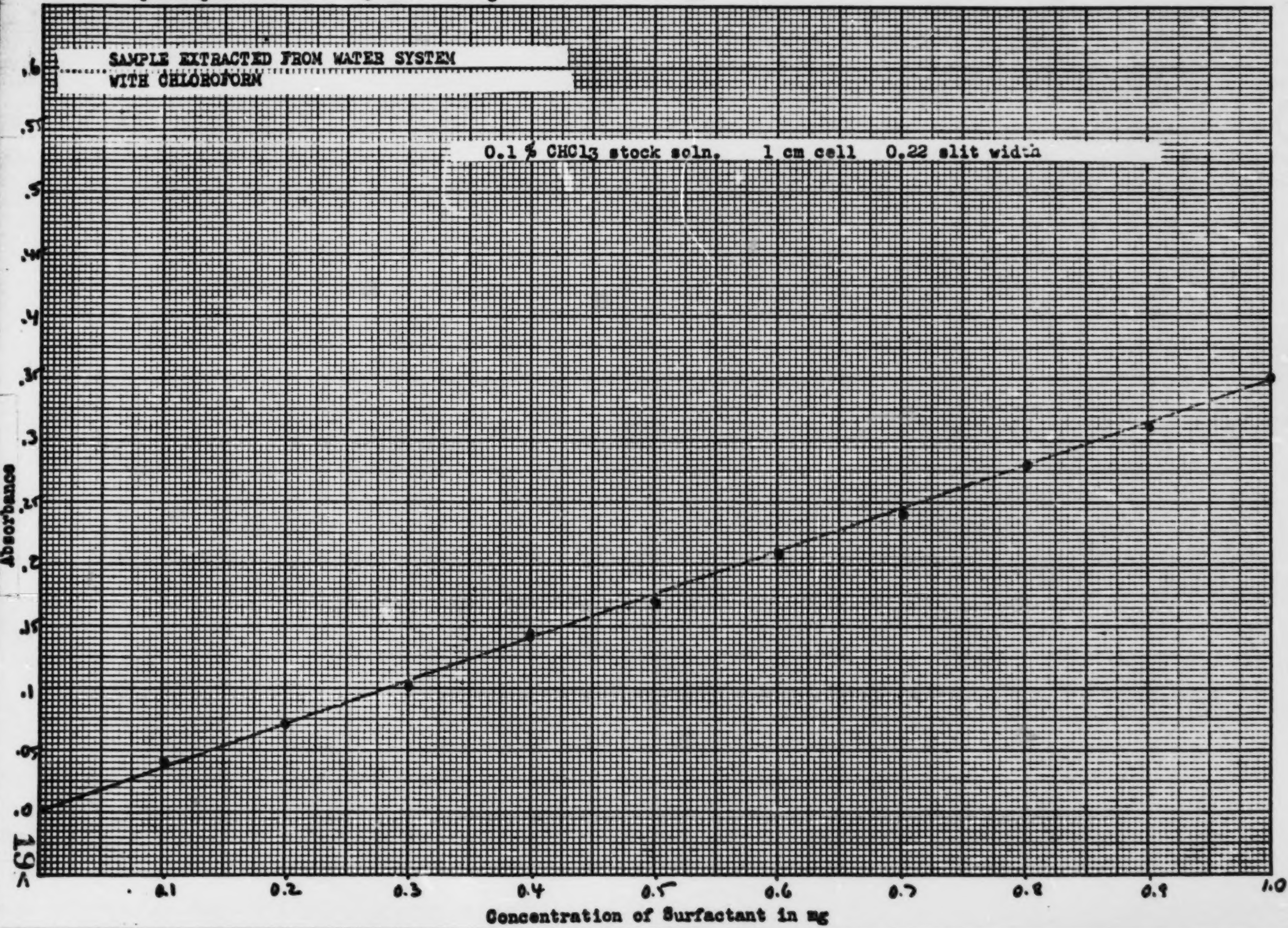

FIGURE 5

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STANDARD CURVE Nonyl Phenol Ethoxylate 9.0 moles EO. Cobalt Thiocyanate Method for Non-Ionic Detergents
Spectrophotometric Analysis of $CHCl_3$ solutions at 318 nm

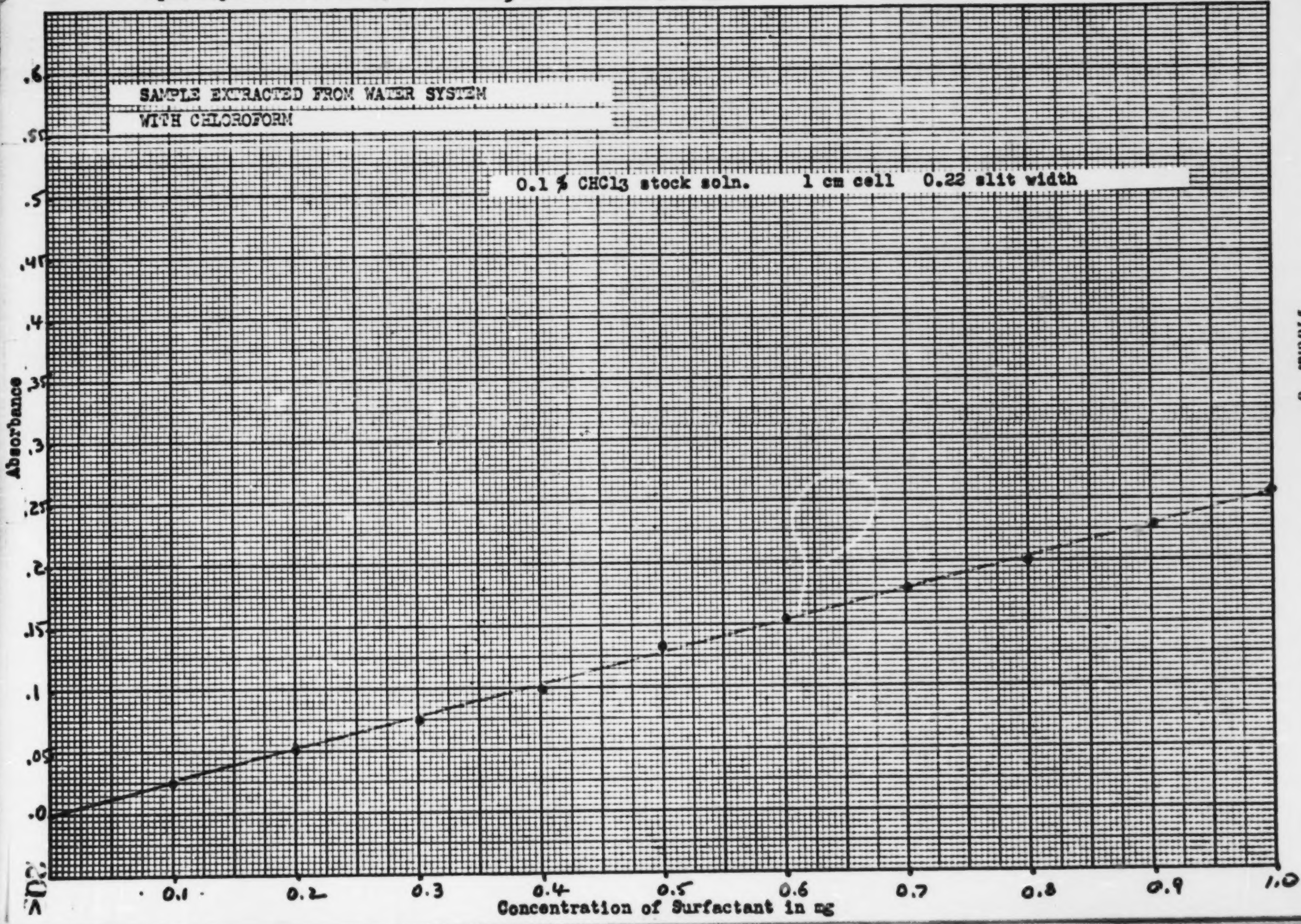

FIGURE 6

Table 7 Potentiometric Titration Data on Unextracted Fatty Alcohol Ethoxylate 30.7 moles EO.

Surfactant Concentration ug	Potentiometric Break Distance in chart units			Volume of Pyrrolidine Dithiocarbamate in ml			Average PBD[1]	Average VPD[2]
	Experiment Number			Experiment Number				
	1	2	3	1	2	3		
50	17	14	16	2.1	2.2	2.2	16	2.2
100	27	26	25	3.6	3.4	3.5	26	3.5
150	36	33	33	5.4	5.2	5.3	34	5.3
200	40	45	47	7.4	7.5	7.8	44	7.6
250	53	54	59	9.0	9.3	9.2	55	9.2
300	66	67	61	10.9	11.0	11.2	65	11.0
400	80	84	84	14.6	14.8	14.6	83	14.7
500	101	100	100	18.0	17.9	17.9	100	17.9
600	117	114	115	22.0	21.8	21.9	115	21.9
700	132	135	138	26.0	25.3	26.6	135	26.0
800	150	158	152	29.4	29.8	29.6	153	29.6
900	170	169	168	33.5	33.4	33.2	169	33.4
1,000	189	187	188	36.9	37.0	37.1	188	37.0

1. PBD = Potentiometric Break Distance
2. VPD = Volume of Pyrrolidine Dithiocarbamate (0.0005 N)

Table 8   Potentiometric Titration Data on Unextracted Fatty Alcohol Ethoxylate 10.6 moles EO.

Surfactant Concentration ug	Potentiometric Break Distance in chart units			Volume of Pyrrolidine Dithiocarbamate in ml			Average PBD[1]	Average VPD[2]
	Experiment Number 1	2	3	Experiment Number 1	2	3		
50	12	11	13	1.5	1.8	1.7	12	1.7
100	18	19	17	2.6	2.4	2.4	18	2.5
150	26	29	28	4.8	4.9	4.8	28	4.8
200	35	35	37	5.7	5.6	5.6	36	5.6
250	41	41	42	7.5	7.4	7.4	41	7.4
300	49	50	51	9.0	8.9	9.0	50	9.0
400	68	65	67	11.7	11.6	11.5	67	11.6
500	81	80	82	14.8	14.7	14.6	81	14.7
600	94	95	95	17.8	17.9	17.7	95	17.8
700	108	109	112	21.2	21.4	21.5	110	21.4
800	125	130	128	24.5	24.7	24.8	128	24.7
900	141	142	143	27.3	27.1	27.4	142	27.3
1,000	154	152	153	30.8	31.0	31.0	153	30.9

1. PBD = Potentiometric Break Distance
2. VPD = Volume of Pyrrolidine Dithiocarbamate (0.0005 N)

Table 9 Potentiometric Titration Data on Unextracted Nonyl Phenol Ethoxylate 9.0 moles EO.

Surfactant Concentration ug	Potentiometric Break Distance in chart units			Volume of Pyrrolidine Dithiocarbamate in ml			Average PBD [1]	Average VPD [2]
	Experiment Number			Experiment Number				
	1	2	3	1	2	3		
50	9	10	9	1.2	1.3	1.3	9	1.3
100	19	17	18	2.6	2.4	2.4	18	2.5
150	26	24	25	3.8	3.6	3.8	25	3.7
200	33	32	32	5.6	5.5	5.4	32	5.5
250	39	38	39	6.7	6.6	6.5	39	6.6
300	46	45	47	8.4	8.5	8.5	46	8.5
400	58	59	57	10.6	10.5	10.6	58	10.6
500	72	70	71	13.8	13.7	13.7	71	13.7
600	88	84	86	16.5	16.4	16.4	86	16.4
700	101	102	100	18.8	18.6	18.6	101	18.7
800	110	111	112	22.0	21.9	22.0	111	22.0
900	125	126	127	24.8	24.5	24.7	126	24.7
1,000	142	140	141	27.7	27.6	27.8	141	27.7

1. PBD = Potentiometric Break Distance
2. VPD = Volume of Pyrrolidine Dithiocarbamate (0.0005 N)

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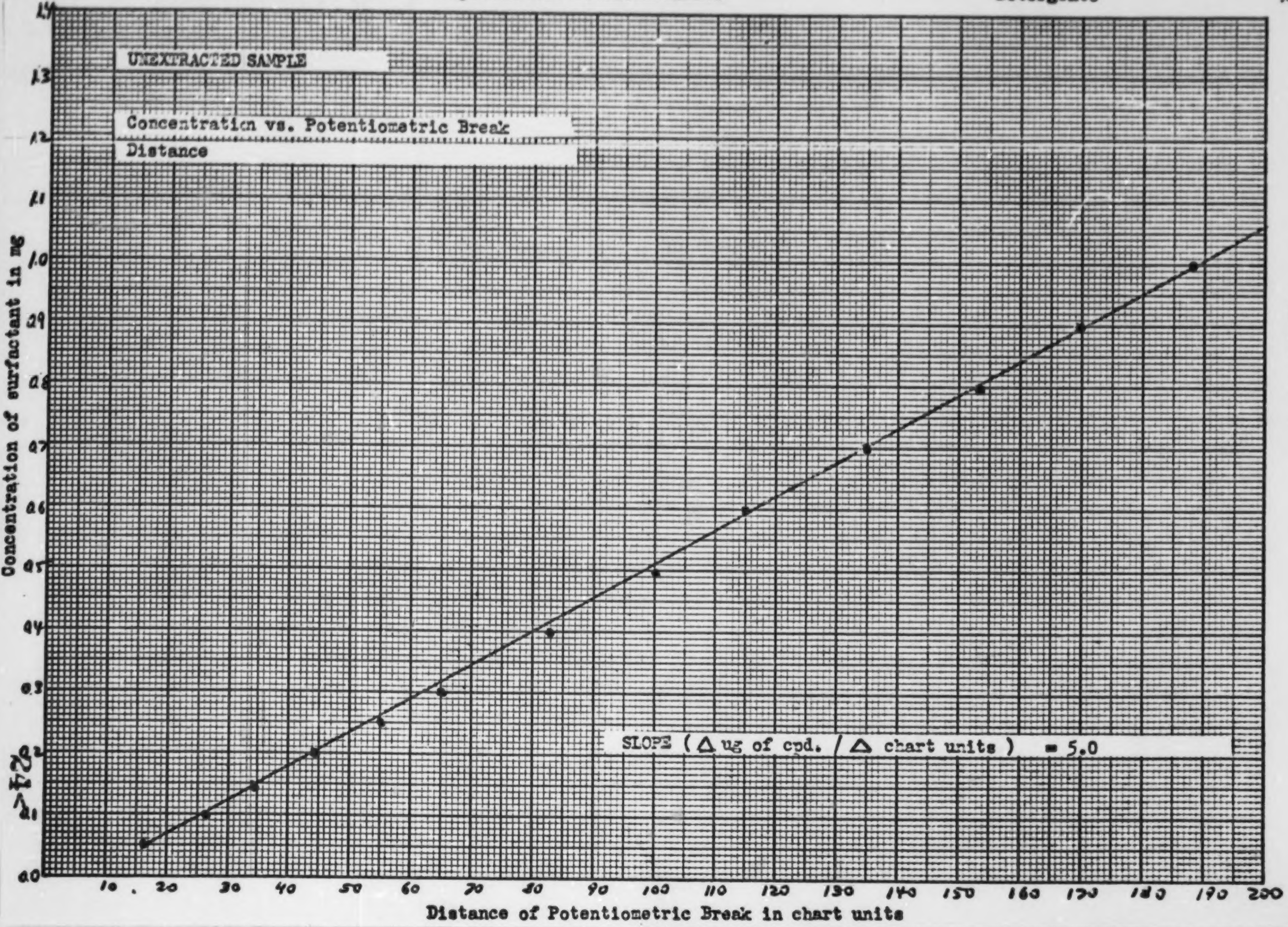
STANDARD CURVE

Fatty Alcohol Ethoxylate 30.7 moles EO.
Potentiometric Titration with Pyrrolidine Dithiocarbamate

Modified Dragendorf Reagent Method for Non-Ionic Detergents

FIGURE 7



K+E 10 X 10 TO ½ INCH 7 X 10 INCHES 46 1323 MADE IN U.S.A.
KEUFFEL & ESSER CO.

STANDARD CURVE

Fatty Alcohol Ethoxylate 10.6 moles EO
Potentiometric Titration with Pyrrolidine Dithiocarbamate

Modified Dragendorf Reagent Method for Non-Ionic Detergents

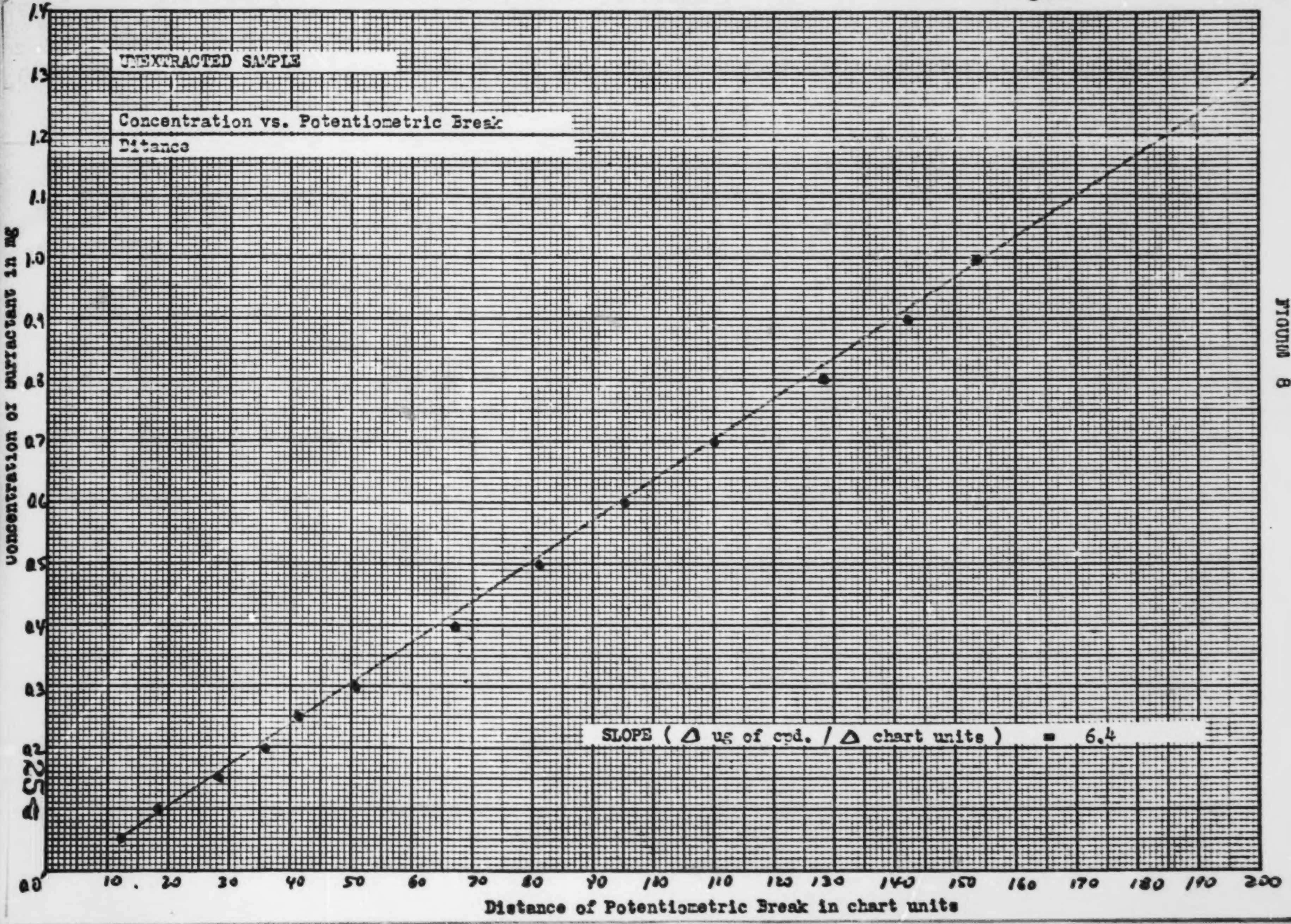


FIGURE 8

FIGURE 9

K•E 10 X 10 TO ½ INCH 46 1323
7 X 10 INCHES MADE IN U.S.A.
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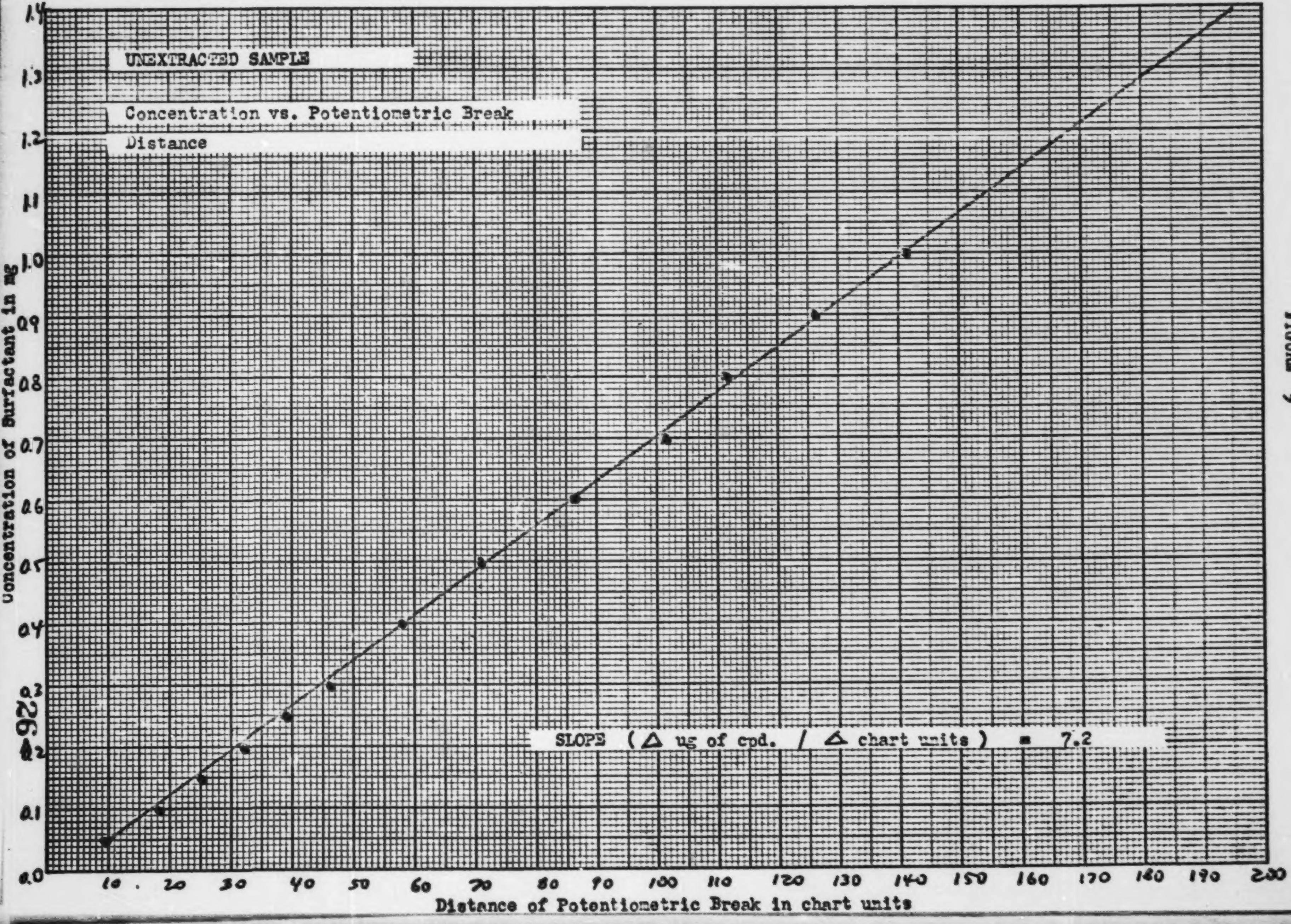
STANDARD CURVE Nonyl Phenol Ethoxylate 9.0 moles EO. Modified Dragendorf Reagent Method for Non-Ionic Detergents
Potentiometric Titration with Pyrrolidine Dithiocarbamate



STANDARD CURVE Fatty Alcohol Ethoxylate 30.7 moles EO. Modified Dragendorf Reagent Method for Non-Ionic Detergents

Potentiometric Titration with Pyrrolidine Dithiocarbamate

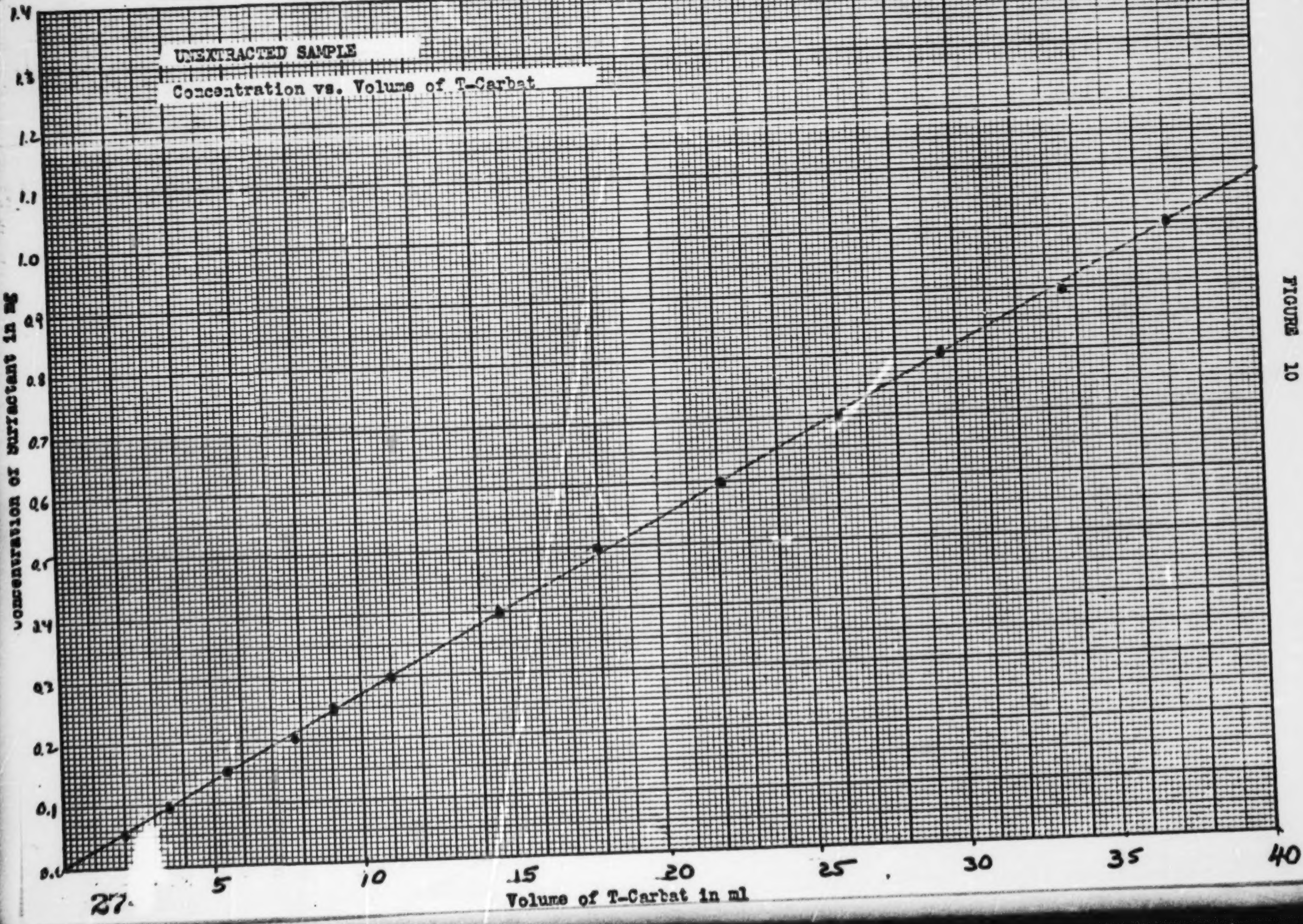


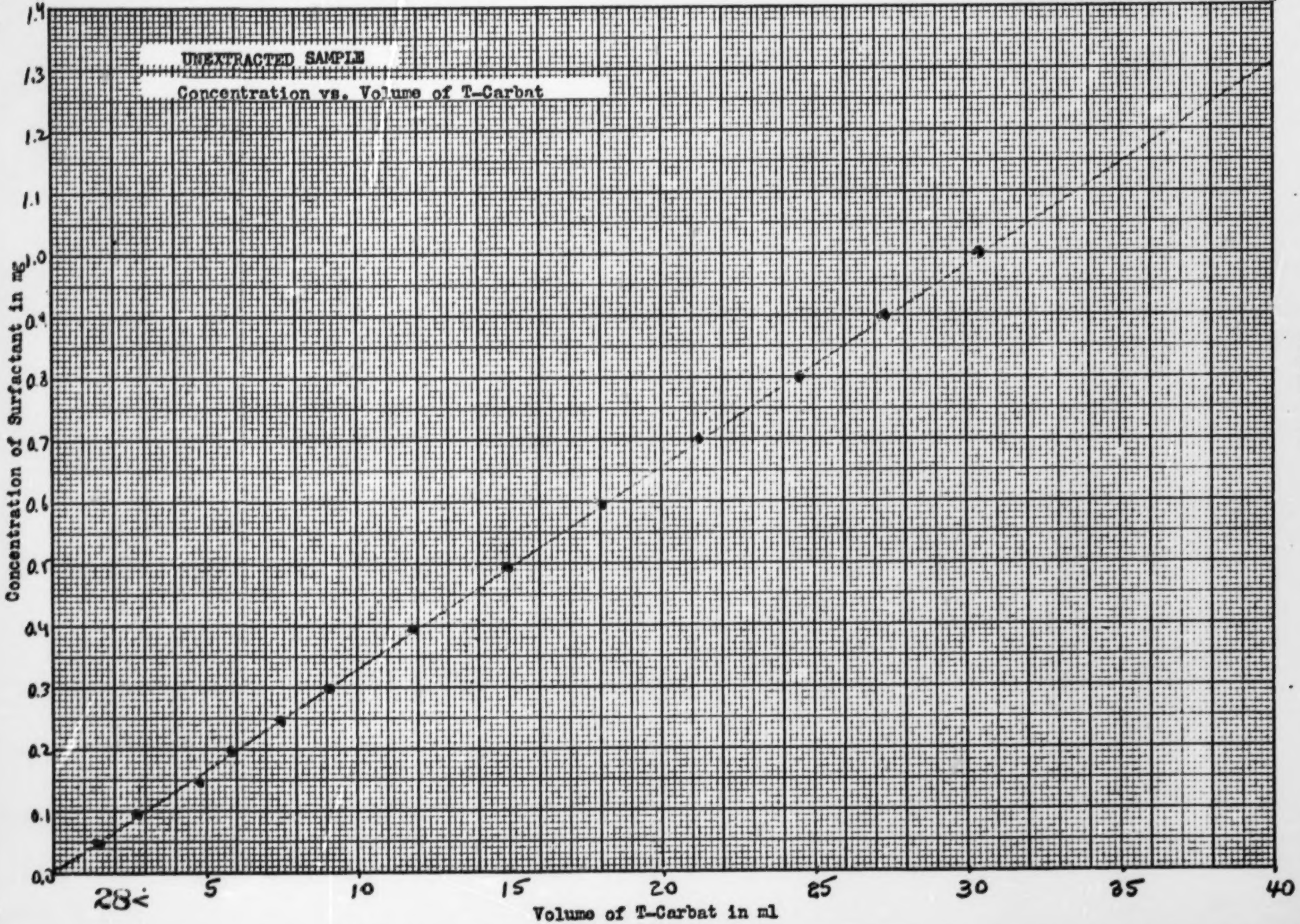
FIGURE 10

K+E 10 X 10 TO ½ INCH 7 X 10 INCHES KEUFFEL & ESSER CO. 46 1323 MADE IN U.S.A.

27

K+E 10 X 10 TO ½ INCH 46 1323
7 X 10 INCHES MADE IN U.S.A.
KEUFFEL & ESSER CO.

STANDARD CURVE Fatty Alcohol Ethoxylate 10.6 moles EO. Modified Dragendorf Reagent Method for Non-Ionic Detergents
Potentiometric Titration with Pyrrolidine Dithiocarbamate



UNEXTRACTED SAMPLE

Concentration vs. Volume of T-Carbat

28<

FIGURE 11

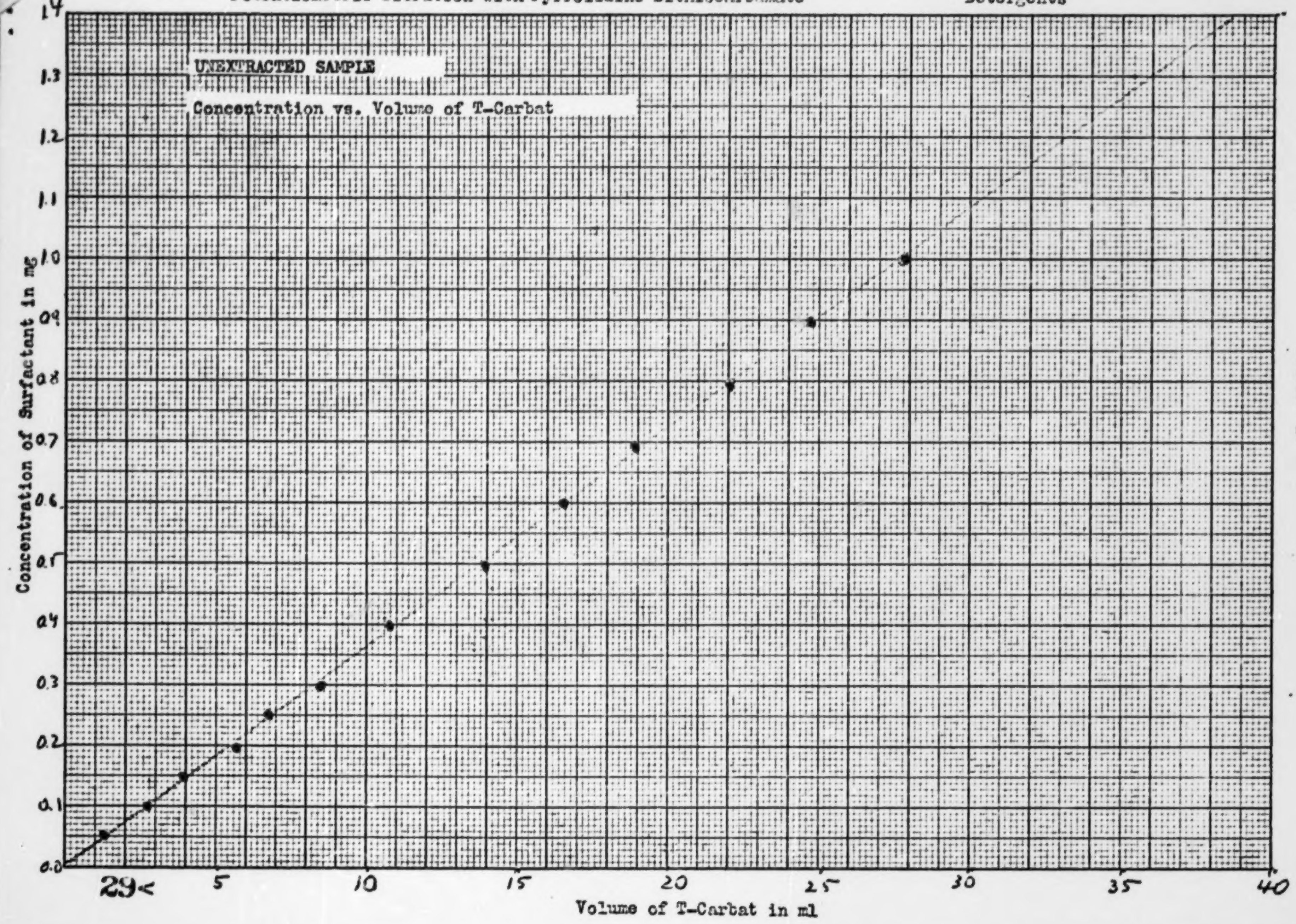
K+E 10 X 10 TO ½ INCH 46 1323
7 X 10 INCHES MADE IN U.S.A.
KEUFFEL & ESSER CO.

STANDARD CURVE

Nonyl Phenol Ethoxylate 9.0 molesEO.

Potentiometric Titration with Pyrrolidine Dithiocarbamate

Modified Dragendorf Reagent Method for Non-Ionic Detergents



UNEXTRACTED SAMPLE

Concentration vs. Volume of T-Carbat

FIGURE 12

Table 10 Potentiometric Titration Data on Fatty Alcohol Ethoxylate 30.7 moles EO. Extracted from Water System

Surfactant Concentration ug	Potentiometric Break Distance in chart units			Volume of Pyrrolidine Dithiocarbamate in ml			Average PBD [1]	Average VPD [2]
	Experiment Number			Experiment Number				
	1	2	3	1	2	3		
50	15	14	15	1.8	1.6	1.6	15	1.7
100	24	23	25	3.3	3.5	3.4	24	3.4
150	34	35	34	5.3	5.4	5.3	34	5.3
200	42	43	44	7.2	7.0	7.0	43	7.1
250	52	53	50	8.6	8.7	8.7	52	8.7
300	61	60	60	10.3	10.5	10.3	60	10.4
400	79	78	76	13.8	14.0	13.9	78	13.9
500	94	93	93	17.2	17.0	17.0	93	17.0
600	109	110	111	21.0	21.4	21.1	110	21.2
700	128	126	127	24.6	24.4	24.4	127	24.5
800	145	142	143	28.2	28.0	27.9	144	28.0
900	161	159	160	32.0	32.2	32.5	160	32.2
1,000	178	176	175	35.2	34.9	35.1	176	35.1

1. PBD = Potentiometric Break Distance
2. VPD = Volume of Pyrrolidine Dithiocarbamate (0.0005 N)

Table 11 Potentiometric Titration Data on Fatty Alcohol Ethoxylate 10.6 moles EO.
Extracted from Water System

Surfactant Concentration ug	Potentiometric Break Distance in chart units			Volume of Pyrrolidine Dithiocarbamate in ml			Average PBD [1]	Average VPD [2]
	Experiment Number			Experiment Number				
	1	2	3	1	2	3		
50	11	12	11	1.4	1.5	1.5	11	1.5
100	17	18	17	2.5	2.3	2.3	17	2.4
150	26	27	26	3.9	4.2	3.8	26	4.0
200	34	33	33	5.9	5.7	5.7	33	5.8
250	39	38	37	6.9	7.0	6.9	38	6.9
300	47	46	46	8.8	8.7	8.6	46	8.7
400	64	63	62	10.9	11.2	11.4	63	11.2
500	76	78	77	13.8	14.0	14.2	77	14.0
600	90	89	91	17.1	17.4	17.0	90	17.2
700	103	102	101	19.8	19.7	20.0	102	19.8
800	120	121	122	22.6	22.9	22.4	121	22.6
900	135	134	133	26.0	25.6	25.7	134	25.8
1,000	146	147	145	28.4	28.6	28.5	146	28.5

1. PBD = Potentiometric Break Distance
2. VPD = Volume of Pyrrolidine Dithiocarbamate (0.0005 N)

Table 12 Potentiometric Titration Data on Nonyl Phenol Ethoxylate 9.0 moles EO. Extracted from Water System

Surfactant Concentration ug	Potentiometric Break Distance in chart units			Volume of Pyrrolidine Dithiocarbamate in ml			Average PBD 1	Average VPD 2
	Experiment Number			Experiment Number				
	1	2	3	1	2	3		
50	8	9	9	1.4	1.3	1.3	9	1.3
100	17	16	18	2.3	2.4	2.3	17	2.3
150	24	23	23	3.5	3.3	3.4	23	3.4
200	30	31	30	5.0	4.9	5.1	30	5.8
250	38	36	37	6.3	6.3	6.7	37	6.4
300	42	43	43	7.4	7.9	7.5	43	7.6
400	56	54	52	9.0	9.4	9.1	54	9.2
500	67	66	69	12.3	12.7	12.5	67	12.5
600	82	81	83	14.7	14.3	14.2	82	14.4
700	96	95	98	17.3	17.7	17.8	96	17.6
800	105	104	102	19.9	20.4	19.6	104	20.0
900	120	119	121	22.6	22.0	22.4	120	22.3
1,000	133	131	132	24.9	25.3	25.0	132	25.1

1. PBD = Potentiometric Break Distance
2. VPD = Volume of Pyrrolidine Dithiocarbamate (0.0005 N)

K+E 10 X 10 TO ½ INCH 46 1323
7 X 10 INCHES MADE IN U.S.A.
KEUFFEL & ESSER CO.

STANDARD CURVE Fatty Alcohol Ethoxylate 30.7 moles EO. Modified Dragendorf Reagent Method for Non-Ionic Detergents
Potentiometric Titration with Pyrrolidine Dithiocarbamate

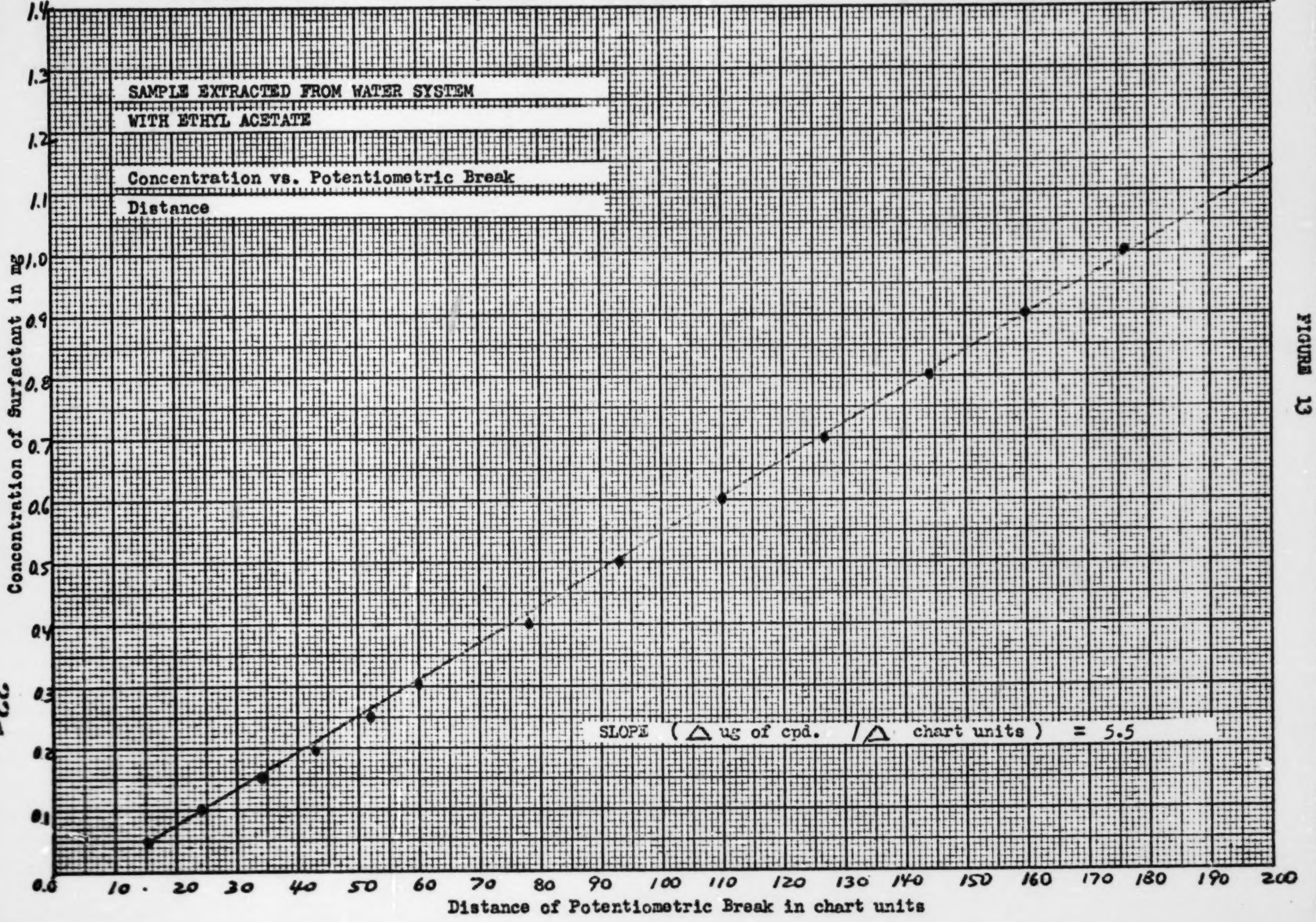


FIGURE 13

K+E 10 X 10 TO ½ INCH 46 1323
7 X 10 INCHES MADE IN U.S.A.
KEUFFEL & ESSER CO.

STANDARD CURVE
Fatty Alcohol Ethoxylate 10.6 moles EO.
Potentiometric Titration with Pyrrolidine Dithiocarbamate
Modified Dragendorf Reagent Method for Non-Ionic Detergents

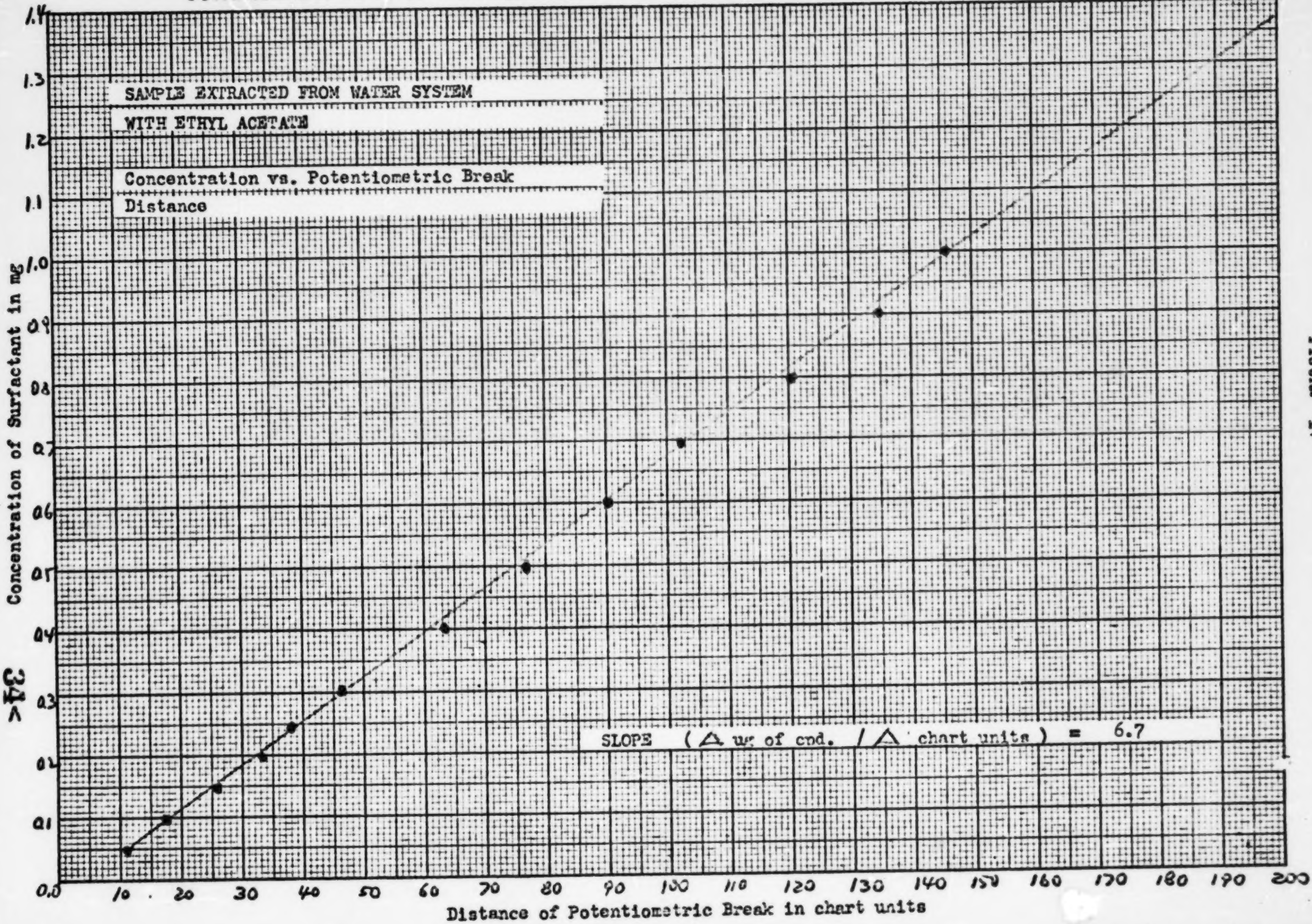


FIGURE 14

K-E 40 1323
KEUFFEL & ESSER CO.

STANDARD CURVE
Nonyl Phenol Ethoxylate 9.0 moles EO.
Potentiometric Titration with Pyrrolidine Dithiocarbamate
Modified Dragendorf Reagent Method for Non-Ionic Detergents

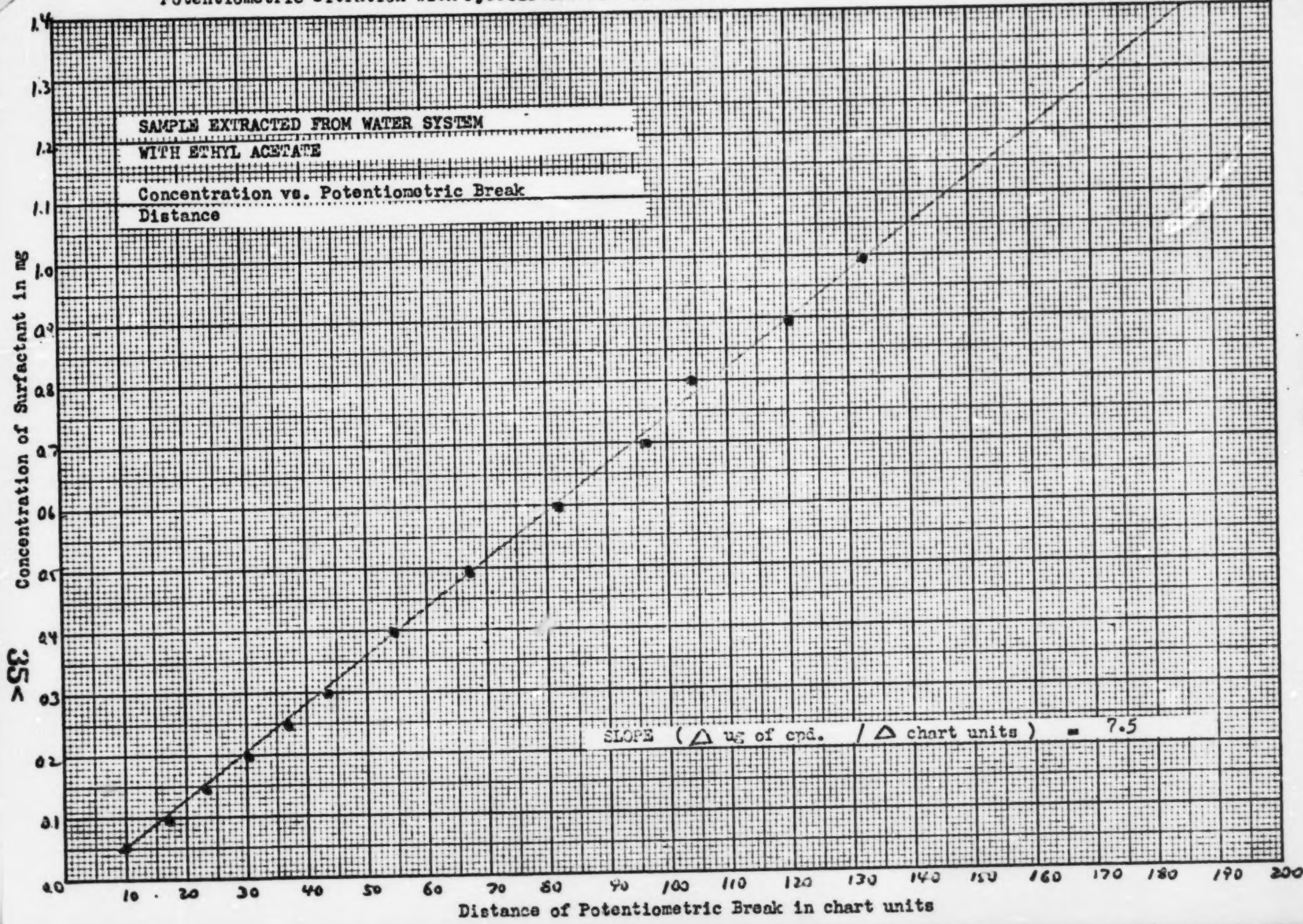


FIGURE 15

K•E 10 X 10 TO ½ INCH 46 1323
7 X 10 INCHES MADE IN U.S.A.
KEUFFEL & ESSER CO.

STANDARD CURVE Fatty Alcohol Ethoxylate 30.7 moles EO. Modified Dragendorf Reagent Method for Non-Ionic Detergents
Potentiometric Titration with Pyrrolidine Dithiocarbamate

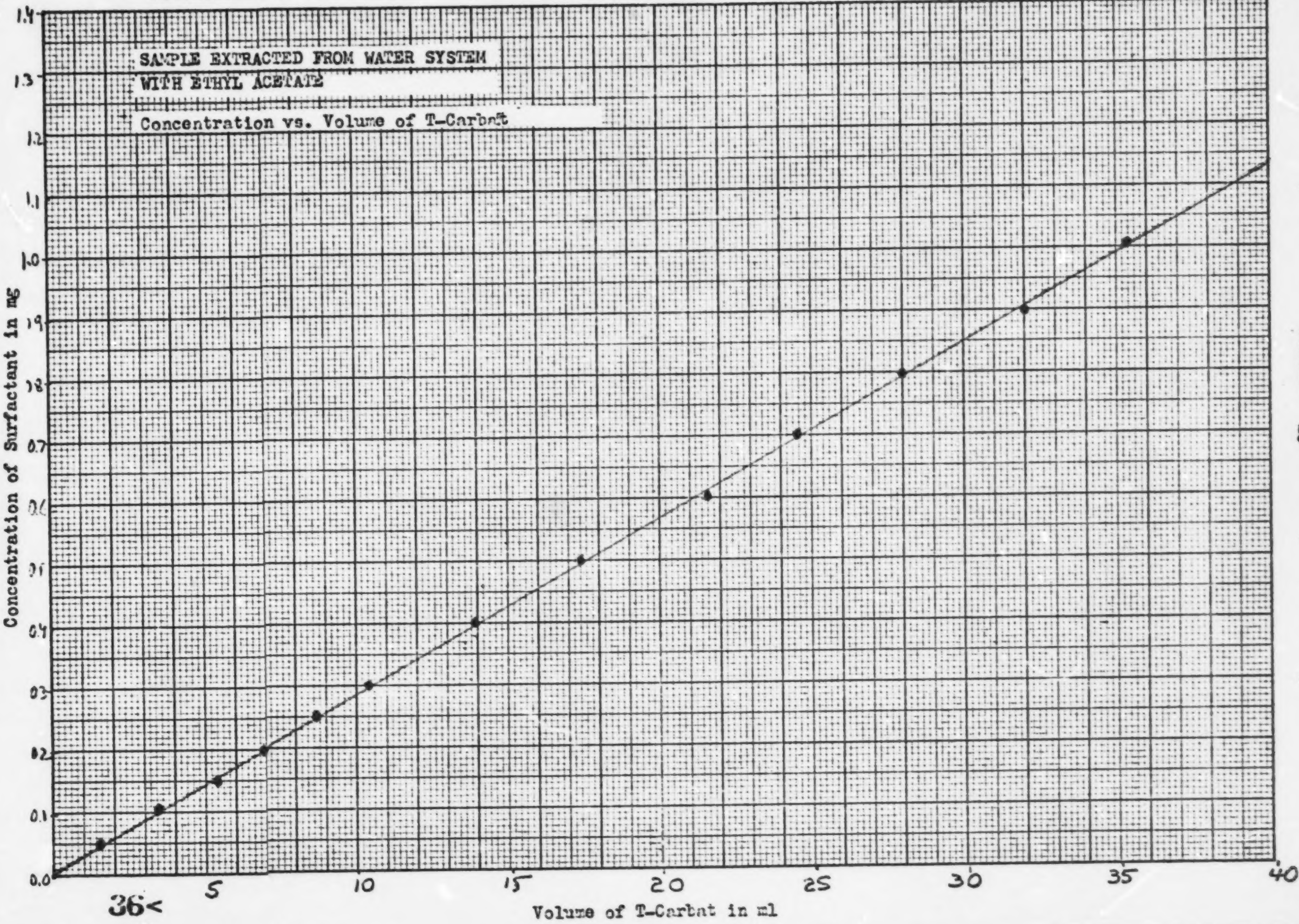
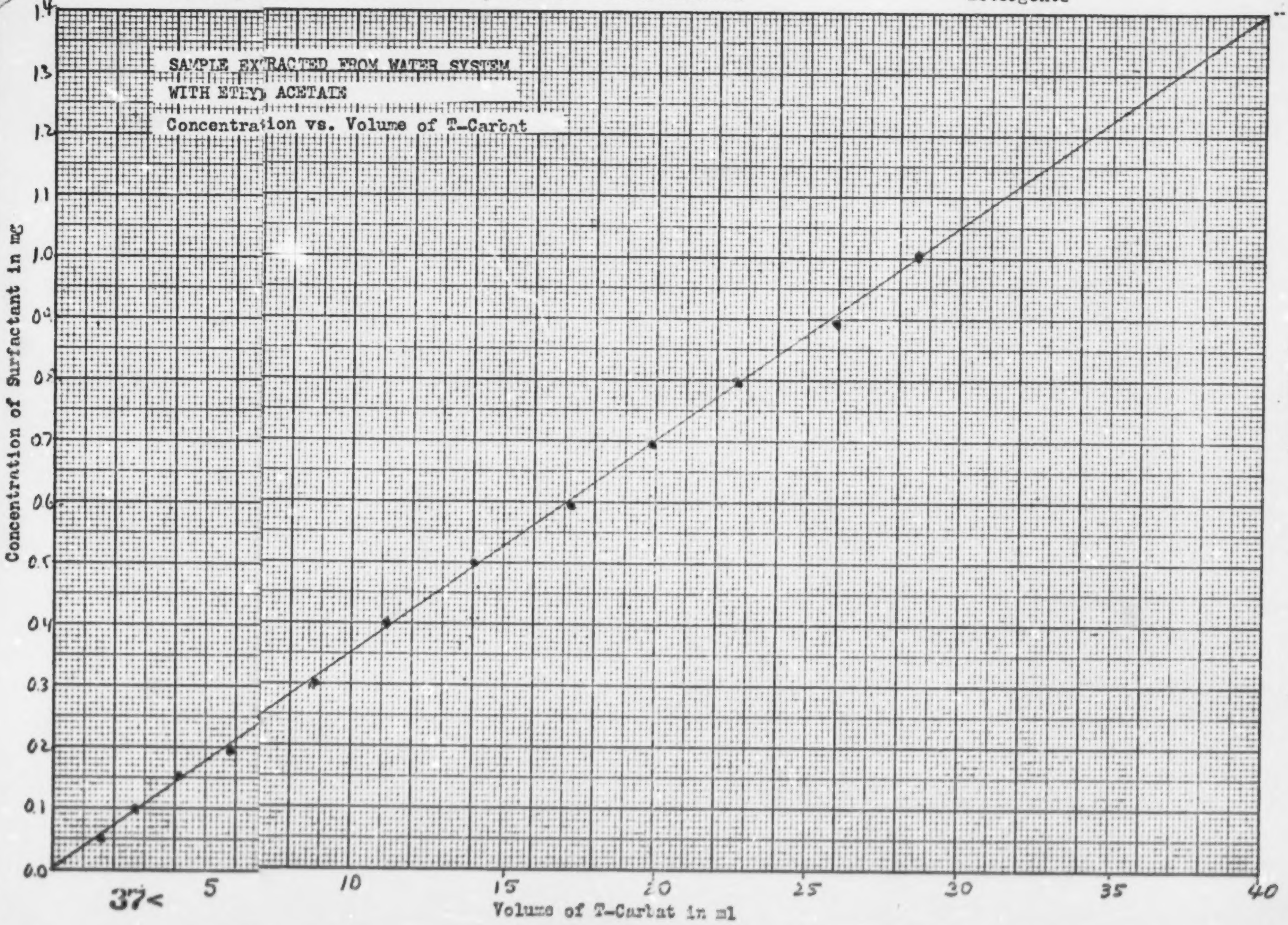


FIGURE 16

FIGURE 17

K+E 10 X 10 TO ½ INCH 46 1323
7 X 10 INCHES
KEUFFEL & ESSER CO.

STANDARD CURVE Fatty Alcohol Ethoxylate 10.6 moles EO. Modified Dragendorf Reagent Method for Non-Ionic Detergents
Potentiometric Titration with Pyrrolidine Dithiocarbamate



37<

K·E 10 X 10 TO ½ INCH 46 1323
7 X 10 INCHES MADE IN U.S.A.
KEUFFEL & ESSER CO.

STANDARD CURVE Nonyl Phenol Ethoxylate 9.0 moles EO. Modified Dragendorf Reagent Method for Non-Ionic Detergents

Potentiometric Titration with Pyrrolidine Dithiocarbamate

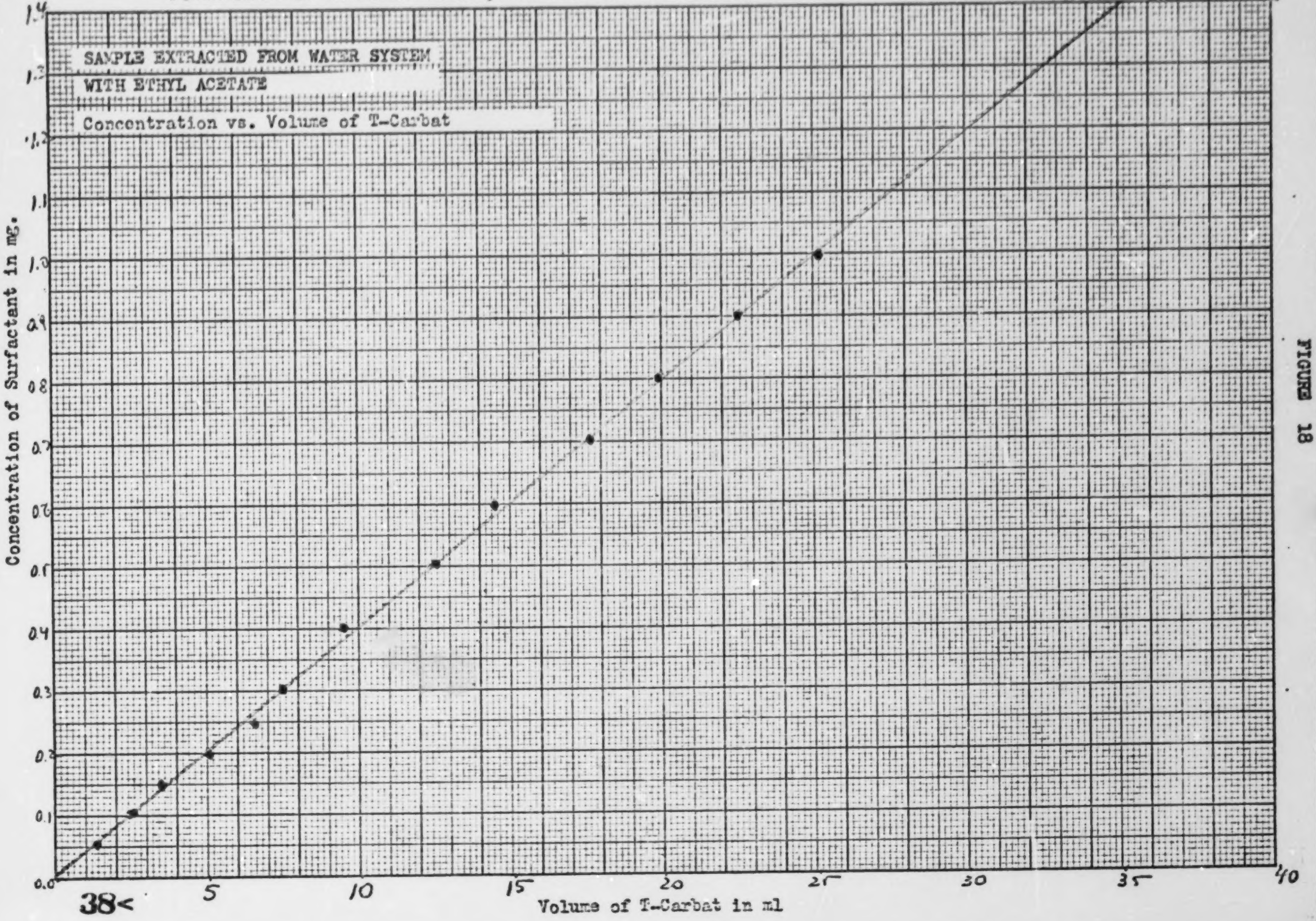


FIGURE 18

38<

Table 13 Potentiometric Titration Data on Fatty Alcohol Ethoxylate 30.7 moles EO. Extracted from Wastewater System

Surfactant Concentration ug	Potentiometric Break Distance in chart units			Volume of Pyrrolidine Dithiocarbamate in ml			Average PBD 1	Average VPD 2
	Experiment Number 1	Experiment Number 2	Experiment Number 3	Experiment Number 1	Experiment Number 2	Experiment Number 3		
50	13	14	12	1.9	1.7	1.8	13	1.8
100	22	21	23	3.2	3.5	3.0	22	3.2
150	32	31	30	5.1	4.8	4.9	31	4.9
200	39	40	41	6.8	7.3	7.1	40	7.1
250	45	44	43	8.5	8.4	8.0	44	8.3
300	57	52	54	10.2	9.8	10.1	54	10.0
400	72	70	73	13.1	13.9	13.7	72	13.6
500	87	86	88	17.5	17.4	17.4	87	17.3
600	105	104	106	21.0	20.5	20.3	105	20.6
700	120	122	121	24.5	24.9	24.7	121	24.7
800	136	138	135	26.8	27.3	27.1	136	27.1
900	152	151	154	30.6	30.8	30.5	152	30.6
1,000	168	167	165	34.3	34.1	34.8	167	34.3

1. PBD = Potentiometric Break Distance
2. VPD = Volume of Pyrrolidine Dithiocarbamate (0.0005 N)

Table 14 Potentiometric Titration Data on Fatty Alcohol Ethoxylate 10.6 moles EO.
Extracted from Wastewater System

Surfactant Concentration ug	Potentiometric Break Distance in chart units			Volume of Pyrrolidine Dithiocarbamate in ml			Average PBD 1	Average VPD 2
	Experiment Number			Experiment Number				
	1	2	3	1	2	3		
50	11	10	10	1.3	1.5	1.4	10	1.4
100	15	15	16	2.9	2.4	2.2	15	2.5
150	22	21	23	4.1	4.2	3.7	22	4.0
200	30	29	28	4.8	5.3	4.9	29	5.0
250	34	34	36	6.3	6.1	6.8	35	6.4
300	43	44	42	7.8	8.3	7.9	43	8.0
400	57	56	57	10.7	10.1	10.1	57	10.3
500	69	70	71	12.4	12.3	12.8	70	12.5
600	81	83	84	15.5	15.6	15.9	83	15.7
700	93	94	95	18.3	17.8	18.0	94	18.0
800	107	110	108	19.8	19.9	20.4	108	20.1
900	121	123	125	23.7	23.4	23.1	123	23.4
1,000	133	134	132	25.6	25.7	25.8	133	25.7

1. PBD = Potentiometric Break Distance
2. VPD = Volume of Pyrrolidine Dithiocarbamate (0.0005 N)

Table 15 Potentiometric Titration Data on Nonyl Phenol Ethoxylate 9.0 moles EO. Extracted from Wastewater System

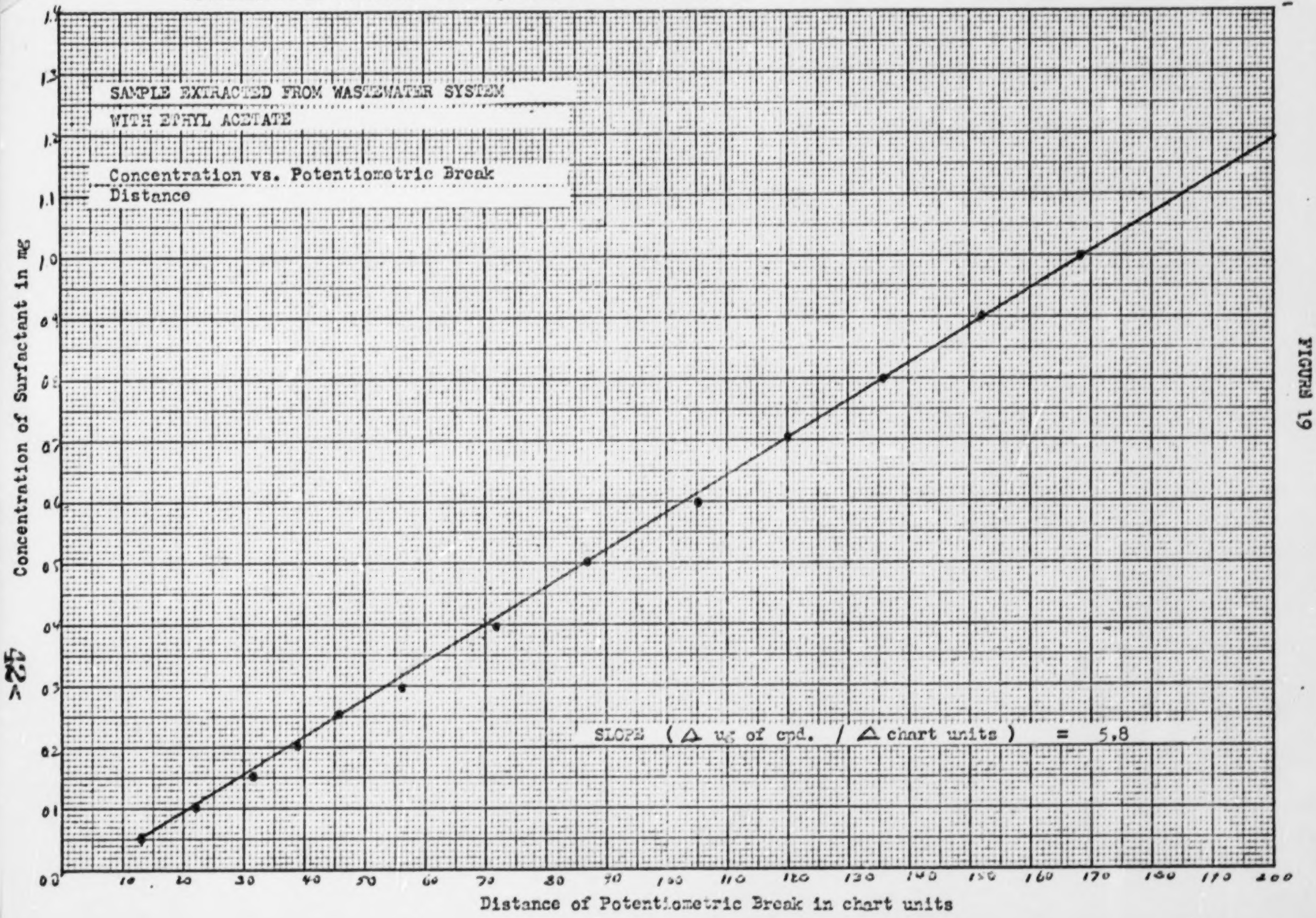
Surfactant Concentration ug	Potentiometric Break Distance in chart units			Volume of Pyrrolidine Dithiocarbamate in ml			Average PBD [1]	Average VPD [2]
	Experiment Number			Experiment Number				
	1	2	3	1	2	3		
50	8	7	8	1.3	1.4	1.0	8	1.2
100	15	16	14	1.9	2.3	1.9	15	2.0
150	21	20	20	3.9	3.2	3.7	20	3.6
200	28	29	27	4.5	4.5	4.2	28	4.4
250	33	35	34	5.1	5.6	5.7	34	5.5
300	41	39	41	7.2	6.7	7.1	40	7.0
400	50	51	51	9.0	9.6	9.7	51	9.4
500	63	60	64	11.6	11.2	11.4	62	11.4
600	77	74	77	13.4	13.6	13.5	76	13.5
700	89	90	88	15.9	16.0	16.3	89	16.1
800	98	97	100	18.6	18.2	18.4	98	18.4
900	110	111	109	20.4	20.2	20.9	110	20.5
1,000	121	121	123	22.9	22.6	23.0	122	22.8

1. PBD = Potentiometric Break Distance
2. VPD = Volume of Pyrrolidine Dithiocarbamate (0.0005 N)

FIGURE 19

K+E 10 X 10 TO ½ INCH 46 1323
7 X 10 INCHES MADE IN U.S.A.
KEUFFEL & ESSER CO.

STANDARD CURVE Fatty Alcohol Ethoxylate 30.7 moles EO. Modified Dragendorf Reagent Method for Non-Ionic Detergents
Potentiometric Titration with Pyrrolidine Dithiocarbamate



K•E 10 X 10 TO ½ INCH 46 1323
7 X 10 INCHES
KEUFFEL & ESSER CO.

STANDARD CURVE Fatty Alcohol Ethoxylate 10.6 moles EO. Modified Dragendorf Reagent Method for Non-Ionic Detergents

Potentiometric Titration with Pyrrolidine Dithiocarbamate

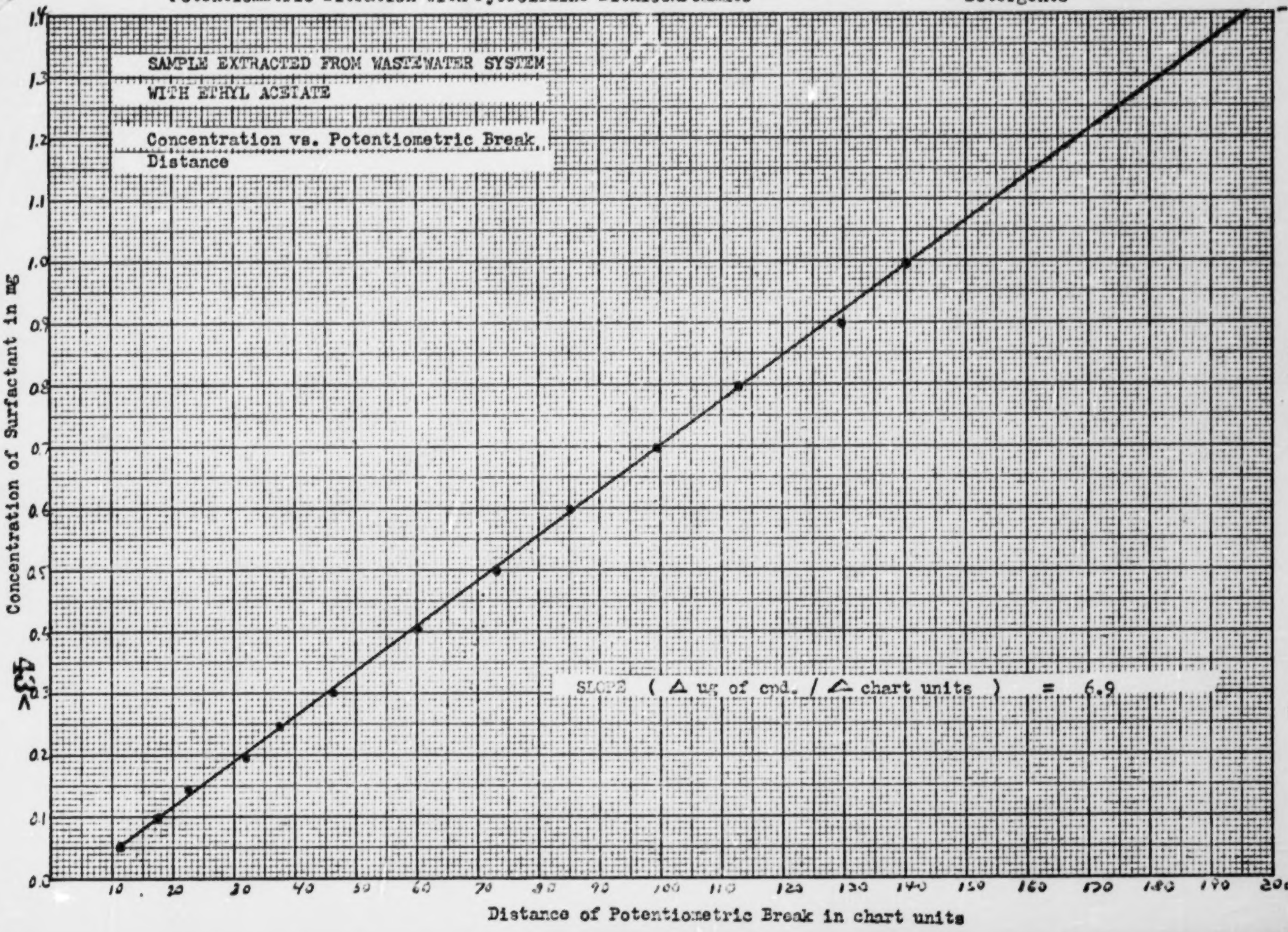
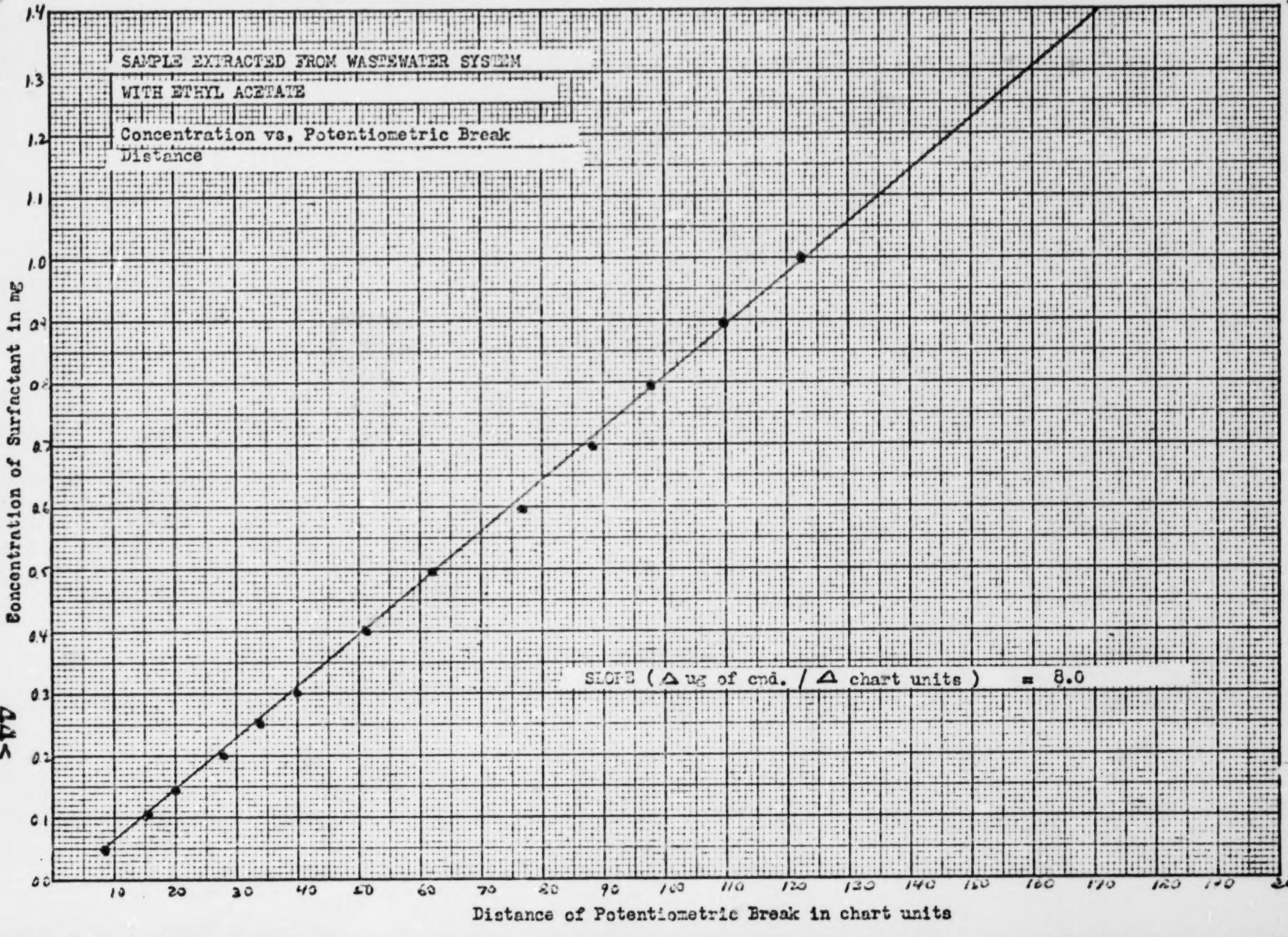


FIGURE 20

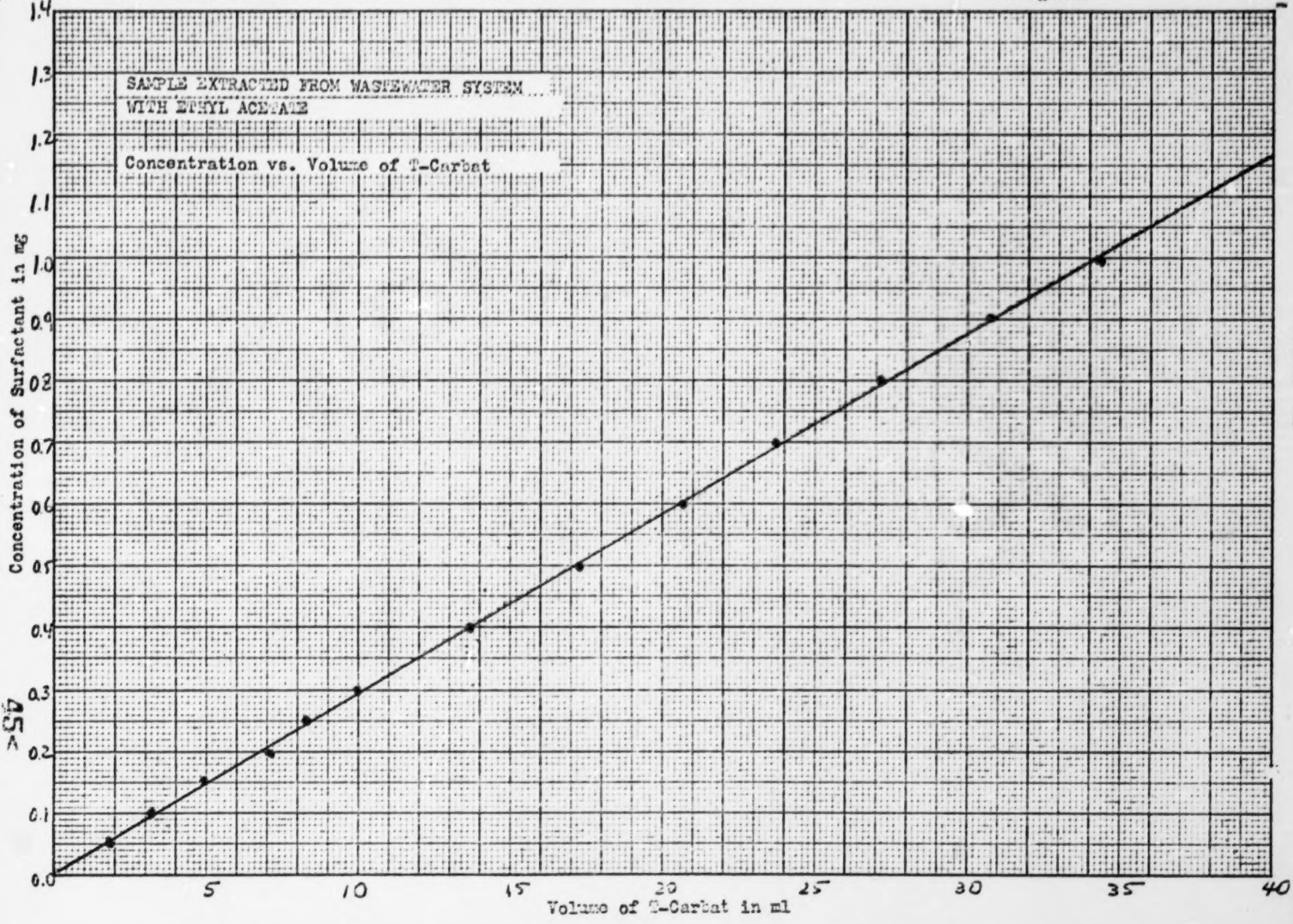
K+E 10 X 10 TO 1/2 INCH 46 1323
7 X 10 INCHES
KEUFFEL & ESSER CO.

FIGURE 21

STANDARD CURVE Nonyl Phenol Ethoxylate 9.0 moles EO. Modified Dragendorf Reagent Method for Non-Ionic Detergents
Potentiometric Titration with Pyrrolidine Dithiocarbamate



STANDARD CURVE Fatty Alcohol Ethoxylate 30.7 moles EO. Modified Dragendorf Reagent Method for Non-Ionic Detergents
Potentiometric Titration with Pyrrolidine Dithiocarbamate



SAMPLE EXTRACTED FROM WASTEWATER SYSTEM WITH ETHYL ACETATE

Concentration vs. Volume of T-Carbat

FIGURE 22

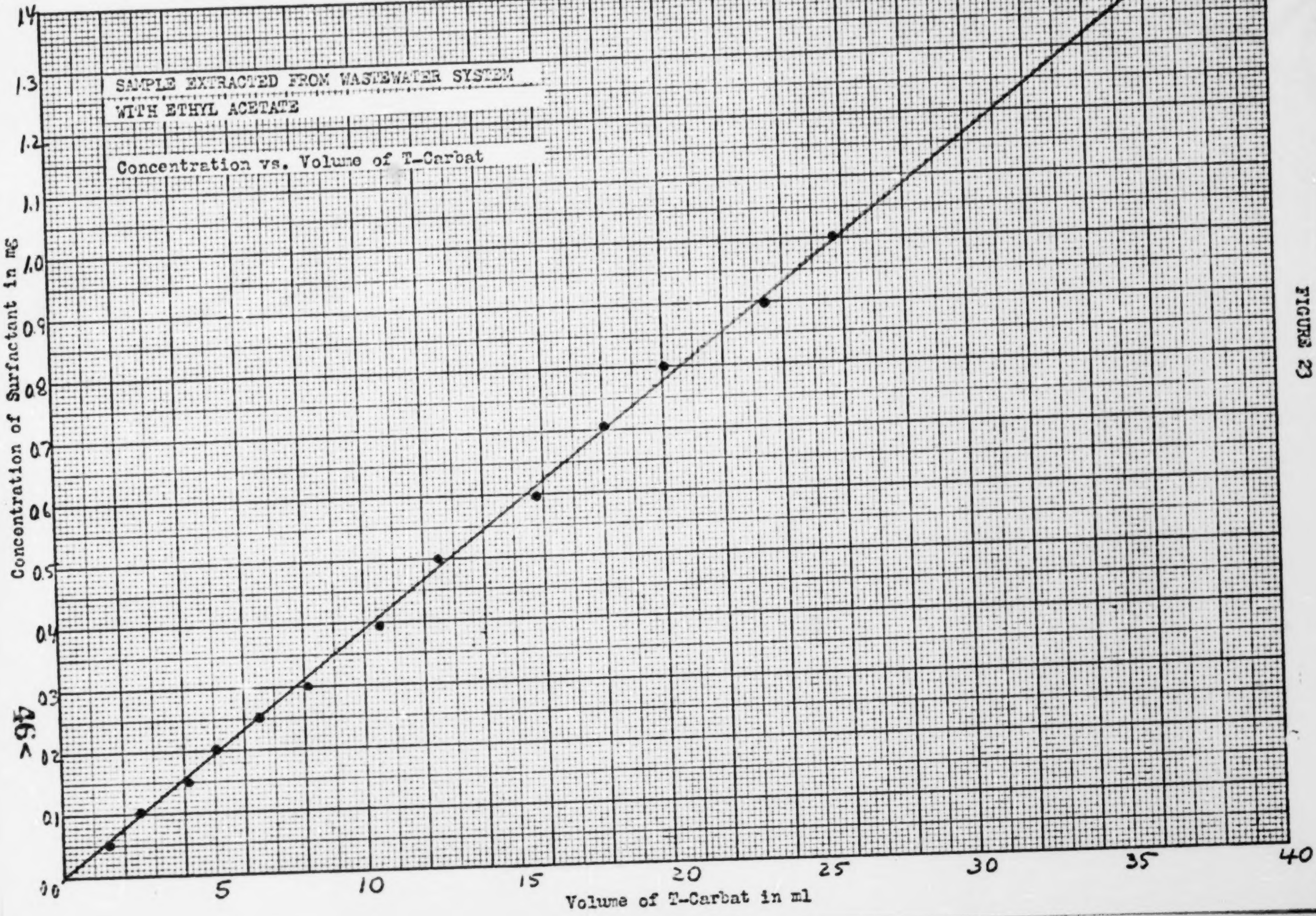
FIGURE 23

K-E 10 X 10 TO ½ INCH 46 1323
7 X 10 INCHES MADE IN U.S.A.
KEUFFEL & ESSER CO.

STANDARD CURVE
Potentiometric Titration with Pyrrolidine Dithiocarbamate

Fatty Alcohol Ethoxylate 10.6 moles EO.

Modified Dragendorf Reagent Method for Non-Ionic Detergents


SAMPLE EXTRACTED FROM WASTEWATER SYSTEM
WITH ETHYL ACETATE

Concentration vs. Volume of T-Carbat

K+E 10 X 10 TO ½ INCH 46 1323
7 X 10 INCHES
KEUFFEL & ESSER CO.

STANDARD CURVE

Nonyl Phenol Ethoxylate 9.0 moles EO.
Potentiometric Titration with Pyrrolidine Dithiocarbamate

Modified Dragendorf Reagent Method for Non-Ionic Detergents

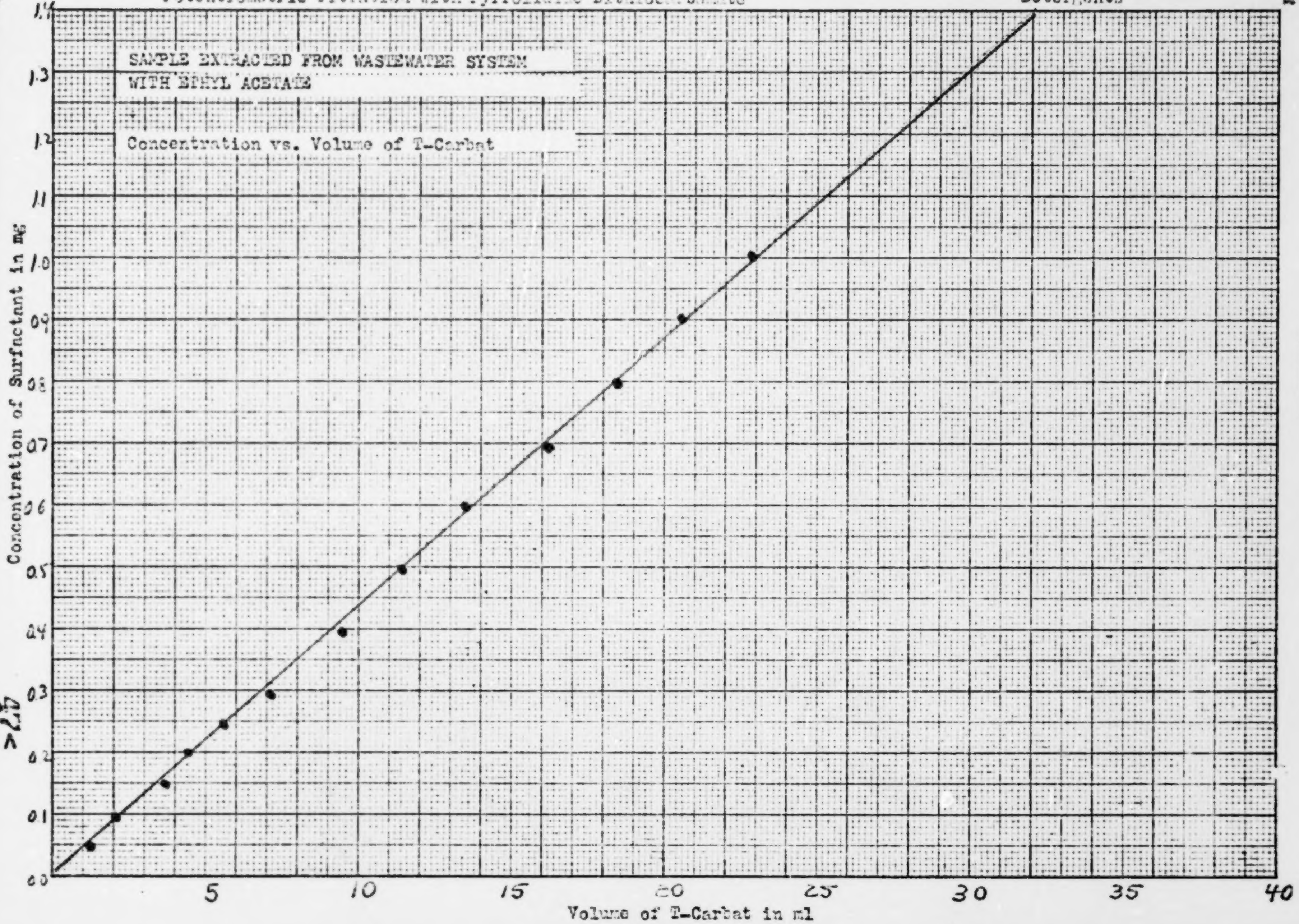


FIGURE 24